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FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* includes those accepted for publication in the ASTM BULLETIN or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1953 is given on the last page of this volume. This supplements the list appearing in the *Proceedings* for 1948 to 1952 covering all special publications published by the Society up to and including 1952.

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SUMMARY OF PROCEEDINGS

OF THE

FIFTY-SIXTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 28–JULY 3, 1953

This summary of the Fifty-sixth Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 28–July 3, 1953, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 34 technical sessions and 1 informal session were held.

The registered attendance of the meeting is as follows: Members present or represented, 1290; committee members, 786; guests, 394; total, 2470; ladies, 450.

The Proceedings are set forth session by session. There were 73 reports and 130 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded:

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[1] The letter ballot on recommendations, affecting standards, distributed to the Society membership, was canvassed on September 15, 1953.

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FIRST SESSION—OPENING SESSION

SUNDAY, JUNE 28, 8:00 P.M.

FORMAL OPENING OF THE FIFTY-SIXTH ANNUAL MEETING, PRESIDENT H. L. MAXWELL

SYMPOSIUM ON METALLIC MATERIALS AT LOW TEMPERATURES[2]

SESSION CHAIRMAN: E. M. MACCUTCHEON
CO-CHAIRMEN: N. A. KAHN AND H. C. BOARDMAN

Introduction—A. L. Tarr.

Brittle Failure in Ships and Other Steel Structures—K. K. Cowart, presented from manuscript by the author.

Analysis of Brittle Behavior in Ship Plates—M. L. Williams, presented from manuscript by the author.

A Critical Survey of Brittle Fracture in Carbon Plate Steel Structures Other Than Ships—M. E. Shank, presented from manuscript by the author.

(*Symposium Continued in Second, Third, Fifth, and Sixth Sessions*)

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[2] To be issued as separate technical publication *ASTM STP No. 158.*

SECOND SESSION—SYMPOSIUM ON METALLIC MATERIALS AT LOW TEMPERATURES

(*Continued from First Session*)

MONDAY, JUNE 29, 9:00 A.M.

SESSION CHAIRMAN: G. W. HOWARD
CO-CHAIRMEN: JAMES O. JACKSON AND WILLIAM M. BALDWIN, JR.

Interest of the Army in Brittle Failures—T. T. Paul, presented from manuscript by the author.

Theory of Brittle Fracture and Criteria for Behavior at Low Temperatures—E. R. Parker, presented from manuscript by the author.

Brittle Fracture: Significance for Engineers—S. L. Hoyt, presented from manuscript by the author.

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented by F. B. Foley, chairman, and accepted as a report of progress.

(*Symposium Continued in Third, Fifth, and Sixth Sessions*)

THIRD SESSION—SYMPOSIUM ON METALLIC MATERIALS AT LOW TEMPERATURES

(*Continued from First and Second Sessions*)

MONDAY, JUNE 29, 2:00 P.M.

SESSION CHAIRMAN: F. B. FOLEY
CO-CHAIRMEN: O. T. MARZKE AND JOHN R. LOW, JR.

Metallurgical Aspects of Low-Temperature Behavior in Ferrous Materials—C. H. Lorig, presented from manuscript by the author.

Fundamentals of Fracture in Metallic Materials—M. Gensamer, presented from manuscript by the author.

The Effect of Size Upon Fracturing—G. R. Irwin, presented from manuscript by the author.

Brittleness, Triaxility, and Localization—W. P. Roop, presented from manuscript by John R. Low, Jr.

Effect of Metallurgical Structure on the Impact Properties of Steels—J. A. Rinebolt, presented from manuscript by the author.

(*Symposium Continued in Fifth and Sixth Sessions*)

FOURTH SESSION—SYMPOSIUM ON FLUORESCENT X-RAY SPECTROGRAPHIC ANALYSIS[3]

MONDAY, JUNE 29, 2:00 P.M.

SESSION CHAIRMAN: E. B. ASHCRAFT

Introduction—E. B. Ashcraft.

Basic Theory and Fundamentals of Fluorescent X-Ray Spectrographic Analysis—Herbert Friedman, La Verne S. Birks, and Edward J. Brooks, presented from manuscript by Mr. Friedman.

General Aspects of Analytical Applications of X-ray Fluorescent Spectrography—J. Sherman, presented from manuscript by the author.

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[3] Issued as separate technical publication *ASTM STP No. 157*.

Examination of Metallic Materials by X-Ray Emission Spectrography—R. M. Brissey, H. A. Liebhafsky, and H. G. Pfeiffer, presented from manuscript by Mr. Liebhafsky.

The Fluorescent X-Ray Spectrographic Analysis of Minerals—Howard F. Carl and William J. Campbell, presented from manuscript by Mr. Campbell.

An Absolute Method of X-Ray Fluorescence Analysis Applied to Stainless Steels—Gordon E. Noakes, presented from manuscript by the author.

Use of Multichannel Recording in X-Ray Fluorescent Analysis—M. F. Hasler and J. W. Kemp, presented from manuscript by C. A. Vaile.

Committee E-2 on Emission Spectroscopy:

Report presented by B. F. Scribner, chairman, and accepted as a report of progress.

Committee E-3 on Chemical Analysis of Metals:

Report presented by Arba Thomas, vice-chairman, in the absence of the chairman, and the following action taken:

Adopted as Standard:

Recommended Practices for Apparatus for Chemical Analysis of Metals (E 50 - 48 T).

FIFTH SESSION—SYMPOSIUM ON METALLIC MATERIALS AT LOW TEMPERATURES

(*Symposium Continued from the First, Second, and Third Sessions*)

MONDAY, JUNE 29, 8:00 P.M.

SESSION CHAIRMAN: IRVING ROBERTS
CO-CHAIRMEN: R. H. RARING AND HAROLD RIND

Evaluation of the Significance of Charpy Tests—William S. Pellini, presented from manuscript by the author.

Significance of V-Notched Impact Test in Evaluation of Armor Plate—A. Hurlich, presented from manuscript by the author.

Notch Bend Tests for Evaluating the Properties of Weldments—R. D. Stout, presented from manuscript by the author.

Reproducibility of Keyhole Charpy and Tear-Test Data on Laboratory Heats of Semikilled Steels—R. H. Frazier, J. W. Spretnak, and F. W. Boulger, presented by Mr. Frazier.

Effect of Specimen Preparation on Notch-Toughness Behavior of Keyhole Charpy Specimens in the Transition Temperature Zone—R. W. Vanderbeck, W. T. Lankford, S. C. Snyder, R. W. Lindsay, and H. D. Wilde, presented by Mr. Vanderbeck.

High Speed Motion Picture Study of Impact Test—H. L. Fry, presented by the author.

(*Symposium Continued in Sixth Session*)

SIXTH SESSION—SYMPOSIUM ON METALLIC MATERIALS AT LOW TEMPERATURES

(*Continued from First, Second, Third, and Fifth Sessions*)

TUESDAY, JUNE 30, 9:00 A.M.

SESSION CHAIRMAN: A. L. TARR
CO-CHAIRMEN: T. N. ARMSTRONG AND JOHN G. THOMPSON

Low-Temperature Impact Properties on Titanium—David E. Driscoll, presented from manuscript by the author.

The Notched Bar Impact Properties of Tempered Martensite in Medium Carbon, Medium Alloy Grades of Steel—M. Baeyertz, W. F. Craig, Sr., and J. P. Sheehan, presented from manuscript by Mr. Sheehan.

Effect of Boron on the Impact Properties of Quenched and Tempered Steels—H. Schwartzbart and J. P. Sheehan, presented from manuscript by Mr. Schwartzbart.

Low-Temperature Brittleness of Steels at High Hardness Levels—Sam Tour, presented from manuscript by the author.

Notch Sensitivity of Steels—E. J. Ripling, presented by the author.

Effect of Carbon and Nitrogen on the Tensile Deformation of High-Purity Iron at 27 C and at −196 C—Lewis D. Hall, presented by title only.

Tension Impact Strength and Strain Distribution at Room and Sub-Zero Temperatures of Stainless and Other Steels—C. R. Mayne, V. N. Krivobok, and C. W. Muhlenbruch presented from manuscript by Mr. Mayne.

Impact Properties of Ferritic Ductile Iron—R. W. Kraft, presented from manuscript by the author.

Ductile and Brittle Failure in Ferritic Nodular Iron—G. N. J. Gilbert, presented from manuscript by James T. MacKenzie.

The Low-Temperature Properties of Cast Iron—G. N. J. Gilbert, presented from manuscript by Mr. T. E. Eagan.

Low-Temperature Toughness of Flake and Spheroidal Graphite Cast Irons—J. S. Vanick, presented from manuscript by the author.

SEVENTH SESSION—SYMPOSIUM ON RADIOACTIVITY IN ASTM WORK

TUESDAY, JUNE 30, 9:00 A.M.

SESSION CHAIRMAN: S. E. EATON

An informal and off-the-record discussion on Radioactivity in ASTM Work consisted of the following presentations:

Introduction—S. E. Eaton.

Welcome and Statement of History, Aims, and Present Activites of Committee E-10 on Radioactive Isotopes—Don M. McCutcheon.

Properties and Uses of Radioisotopes—G. D. Calkins.

Applications of Radioactive Measurements to ASTM Work—O. M. Elliott.

Design of Radioisotope Laboratories—G. G. Manov and O. M. Bizzell.

Training Personnel in Radioisotope Techniques—Donald R. Smith.

Instrumentation—E. H. Wakefield.

Management Problems Resulting from Radioisotope Utilization by Industry—W. L. Davidson.

[While these were intended as informal presentations, the committee is giving consideration to having papers on individual subjects prepared looking towards the publication of a Symposium on Radioactivity in ASTM Work.[3a]]

(*Symposium Continued in Twelfth Session*)

EIGHTH SESSION—REPORT SESSION

TUESDAY, JUNE 30, 11:30 A.M.

SESSION CHAIRMAN: A. C. FIELDNER

Committee D-3 on Gaseous Fuels:

Report presented by K. R. Knapp, secretary, in the absence of the chairman, and the following actions taken:

Accepted as Tentative:

Method of Sampling Liquefied Petroleum Gases (D 1265 – 53 T) (Jointly with Committee D-2 on Petroleum Products and Lubricants.)

Adopted as Standard:

Method for Analysis of Natural Gases by the Volumetric Chemical Method (D 1136 – 50 T)

Method for Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 – 50 T)

Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142 – 50 T)

Method for Sampling Natural Gas (D 1145 – 50 T)

[3a] Will be issued as separate publication, entitled, "Symposium on Radioactivity—Isotopes and Tracer Techniques," *ASTM STP No. 159.*

Committee D-5 on Coal and Coke:

Report presented by W. W. Anderson, chairman, and the following action taken:

Adopted as Standard:

Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances (D 980 – 48 T)

Committee D-7 on Wood:

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

Accepted as Tentative:

Spec. for Copperized Chromated Zinc Chloride (D 1271 – 53 T) With editorial revisions in Section 2 to present more correctly the composition and properties.
Spec. for Pentachlorophenol (D 1272 – 53 T)
Methods for Chemical Analysis of Copperized Chromated Zinc Chloride (D 1273 – 53 T)
Methods for Chemical Analysis of Pentachlorophenol (D 1274 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Round Timber Piles (D 25 – 52 T)

Adopted as Standard:

Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 – 50 T)

Adopted as Standard, Revisions in:

Spec. for Creosote (D 390 – 49)
Spec. for Creosote-Coal Tar Solution (D 391 – 50)

Committee D-10 on Shipping Containers:

Report presented by G. E. Falkenau, chairman, and the following actions taken:

Accepted as Tentative:

Test for Water Vapor Permeability of Shipping Containers by Cycle Method (D 1276 – 53 T)

Adopted as Standard:

Methods of Testing Large Shipping Cases and Crates (D 1083 – 50 T)

Committee D-21 on Wax Polishes and Related Materials:

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

Accepted as Tentative:

Test for Nonvolatile Matter (Total Solids) in Water-Emulsion Waxes (D 1289 – 53 T)
Test for Sediment in Water-Emulsion Waxes by Means of Centrifuge (D 1290 – 53 T)
Test for Total Ash and Silica in Water-Emulsion Waxes (D 1288 – 53 T)

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented by W. W. Becker, secretary, and accepted as a report of progress.

NINTH SESSION—REPORT SESSION

TUESDAY, JUNE 30, 11:30 A.M.

SESSION CHAIRMAN: G. R. GOHN

Committee B-1 on Wires for Electrical Conductors:

Report presented by D. Halloran, chairman, and the following actions taken:

Accepted as Tentative:

Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire-Fabrication (B 279 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Hard-Drawn Copper Wire (B 1 – 52 T)
Spec. for Soft or Annealed Copper Wire (B 3 – 52 T): revising the second paragraph of Section 7 (*a*) to read "If the average is more than the tensile strength in Table I minus 800 psi and the tensile strength of each of the individual specimens is equal to or less than the value in Table I, six additional specimens from six production units, other than the four originally sampled, shall be tested. The lot shall be considered conforming if the tensile strength of each of the ten specimens is not more than the appropriate tensile strength value in Table I and the average of the ten specimens is not more than that value minus 800 psi. The lot shall be considered to have

failed to meet the tensile strength requirement if any of the ten specimens exceeds the appropriate tensile strength value in Table I or if the average of the ten specimens exceeds that value minus 800 psi."

The second paragraph of Section 7 (*b*) was also revised to read "If the average is less than the elongation in Table I plus 2.5 per cent and the elongation of each of the individual specimens is equal to or more than the value in Table I, six additional specimens from six production units, other than the four originally sampled, shall be tested. The lot shall be considered conforming if the elongation of each of the ten specimens is not less than the appropriate elongation value in Table I, and the average of the ten specimens is not less than that value plus 2.5 per cent. The lot shall be considered to have failed to meet the elongation requirement if any of the ten specimens is less than the appropriate elongation value in Table I or if the average of the ten specimens is less than that value plus 2.5 per cent."

Spec for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 52T)

Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (B 172 – 52 T)

Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members for Electrical Conductors (B 172 – 52 T)

Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 52 T)

Spec. for Lead-Coated and Lead-Alloy Coated Soft Copper Wire for Electrical Purposes (B 189 – 52 T)

Spec. for Hard-Drawn Aluminum Wire for Electrical Purposes (B 230 – 52 T)

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 – 52 T)

Adopted as Standard, Revisions in:

Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 52)

Spec. for Bronze Trolley Wire (B 9 – 52)

Spec. for Hard-Drawn Copper Alloy Wires for Electrical Conductors (B 105 – 52)

Spec. for Concentric-Lay-Stranded Auminum Conductors, Hard-Drawn (B 231 – 52)

Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for High-Resistivity, Low-Temperature Coefficient Wire (B 267 – 52 T)

Adopted as Standard:

Spec. for 17 per cent Chromium-Iron Alloy for Sealing to Glass (B 256 – 51 T)

Spec. for 28 per cent Chromium-Iron Alloy for Sealing to Glass (B 257 – 51 T)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by G. H. Harnden, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Seamless Copper Tube for Refrigeration Service (B 280 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Wrought Copper and Copper-Alloy Plate Sheet, Strip, and Rolled Bar (B 248 – 52 T)

Spec. for Seamless Brass Boiler Tubes (B 14 – 49)

Spec. for Naval Brass Rod, Bar, and Shapes (B 21 – 52)

Spec. for Seamless Red Brass Pipe, Standard Sizes (B 43 – 52)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 52)

Spec. for Gilding Metal Strip (B 130 – 52)

Spec. for Gilding Metal Bullet Jacket Cups (B 131 – 52)

Spec. for Manganese Bronze Rod, Bar, and Shapes (B 138 – 52)

Spec. for Aluminum Bronze Rod, Bar, and Shapes (B 150 – 52)

Spec. for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 52)

Spec. for Copper-Alloy Condenser Tube Plates (B 171 – 52)

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative:

Recommended Practice for the Preparation of Copper and Copper-Base Alloys for Electroplating (B 281 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 45 T)

Spec. for Electrodeposited Coatings of Lead on Steel (B 200 – 45 T)

Adopted as Standard:

Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 - 51 T)
Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 - 51 T)
Recommended Practice for Preparation of Zinc Base Die Castings for Electroplating (B 252 - 51 T)
Recommended Practice for Preparation of and Electroplating on Stainless Steel (B 254 - 51 T)
Recommended Practice for Preparation of and Electroplating on Aluminum Alloys (B 253 - 51 T), as revised

Adopted as Standard, Revisions in:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc Base Die Castings (B 142 - 51)
Recommended Practice for Preparation of High Carbon Steel for Electroplating (B 242 - 51)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by F. V. Lenel, chairman, and the following action taken:

Accepted as Tentative:

Spec. for Sintered Metal Powder Structural Parts from Brass (B 282 - 53 T)

Committee C-3 on Chemical-Resistant Mortars:

Report presented in the absence of the chairman by F. O. Anderegg, and the following actions taken:

Accepted as Tentative:

Test for Compressive Strength of Resin-Type Chemical-Resistant Mortars (C 306 - 53 T)
Test for Tensile Strength of Resin-Type Chemical-Resistant Mortars (C 307 - 53 T)
Test for Working and Setting Times of Resin-Type Chemical-Resistant Mortars (C 308 - 53 T)

Committee C-4 on Clay Pipe:

Report presented by J. C. Riedel, chairman, and accepted as a report of progress.

TENTH SESSION—LUNCHEON, PRESENTATION OF AWARDS OF MERIT, INTRODUCTION OF NEW OFFICERS, PRESIDENT'S ADDRESS, AWARD OF HONORARY MEMBERSHIPS, REPORT OF BOARD OF DIRECTORS, RECOGNITION OF 50- AND 40-YEAR MEMBERS

TUESDAY, JUNE 30, 12:00 A.M.

CHAIRMAN: PAST-PRESIDENT T. S. FULLER

About 300 were present at this annual luncheon session, including a number of ladies and visitors.

Awards of Merit:

The Chairman of the 1953 Award of Merit Committee, H. M. Hancock, read brief citations and presented the following to President Maxwell, who conferred on them, on behalf of the Board of Directors, the certificate of the Award of Merit:

Everett G. Ham
James T. MacKenzie
Dalton G. Miller
R. E. Peterson
Carlton H. Rose
W. A. Selvig
E. I. Shobert II
Earl R. Stivers
Jerome Strauss
Roderick B. Young

Election of Officers:

Results of the letter ballot on election of new officers were announced by the Chairman of the Tellers' Committee, W. J. McCoy. The results were as follows:

For President, to serve for one year:
  L. C. Beard, 1204 votes
For Vice-President, to serve for two years:
  C. H. Fellows, 1205 votes
For Directors, to serve for three years:
  N. A. Fowler, 1200 votes
  R. T. Kropf, 1199 votes
  T. F. Olt, 1202 votes
  J. R. Townsend, 1193 votes
  K. B. Woods, 1201 votes

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect Beard and Vice-President Fellows, the latter two men responding briefly.

Presidential Address:

The annual President's Address was presented by Harold L. Maxwell, his subject being “People and Things.” This address is printed in the July ASTM BULLETIN.

Award of Honorary Memberships:

The Executive Secretary read citations for the following two members who had been elected by the Board of Directors to Honorary Membership in the Society:

Lloyd B. Jones (*Deceased*)
Horace H. Lester

Dr. Maxwell presented the certificate to Dr. Lester. Mr. Jones' son Charles received the certificate for his father.

Report of the Board of Directors:

R. J. Painter, Executive Secretary, presented a brief résumé of the Report of the Board of Directors.

Recognition of 50- and 40-year Members, Special Presentations:

The Executive Secretary read the names of nine members, both individual and company, who had been continuously affiliated with the Society for 50 years. Certificates to that effect were presented. The procedure was repeated for thirty-seven 40-year members. A complete list of the 50- and 40-year members appears in the July, 1953 ASTM BULLETIN.

Chairman Fuller then presented the Past President's pin to retiring President Harold H. Maxwell. Past President J. G. Morrow as the Senior Past-President, retiring from the Board of Directors, was presented. He had completed eight years of service on the Board of Directors.

ELEVENTH SESSION—REPORT SESSION

TUESDAY, JUNE 30, 2:30 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee A-2 on Wrought Iron:

Report presented by A. D. Morris, chairman, and accepted as a report of progress.

Committee A-3 on Cast Iron:

Report presented by D. E. Krause, secretary, in the absence of the chairman, and the following actions taken:

Accepted as Tentative:

Methods of Chill Testing of Cast Iron (A 367 - 53 T)

Accepted as Tentative, Revisions in:

Spec. for Automotive Gray Iron Castings (A 159 - 49 T)

Adopted as Standard:

Spec. for Gray Iron Castings for Pressure Containing Parts for Temperatures up to 650 F (A 278 - 51 T)
Spec. for Gray Iron Castings for Elevated Temperatures for Non-Pressure Containing Parts (A 319 - 48 T)

Committee A-6 on Magnetic Properties:

Report presented by R. L. Sanford, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Methods of Testing Magnetic Materials (A 34 – 49)

Test for Permeability of Feebly Magnetic Materials (A 342 – 49)

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and accepted as a report of progress.

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by B. W. Gonser, chairman, and accepted as a report of progress.

Joint Committee on Filler Metal:

Report presented in the absence of the chairman by J. W. Caum, and accepted as a report of progress.

TWELFTH SESSION—SYMPOSIUM ON RADIOACTIVITY IN ASTM WORK

(*Continued from Seventh Session*)

TUESDAY, JUNE 30, 2:15 P.M.

SESSION CO-CHAIRMEN: G. G. MANOV AND E. B. ASHCRAFT

Atomic Energy Commission Movie on Methodology—G. G. Manov.

Demonstration Period.

- Absorption and Backscatter Types of Beta Ray Thickness Gages—J. Pennock, Tracerlab, Inc.
- Radioisotopes in Radiography—D. T. O'Connor, Naval Ordnance Laboratories.
- Beta Ray Hydrogen Carbon Ratio Analyzer—R. B. Jacobs, Standard Oil Company of Indiana and H. M. Sullivan, Central Scientific Co.
- Radiolabeled Dirt in Cleaning Tests—O. K. Neville, Nuclear Instrument and Chemical Corp.

Panel Discussion:

The panel consisted of the previous speakers with other selected ASTM committee members. The purpose was to give other ASTM committees a chance to tell of their activities, plans, or thoughts, on using radioactivity and for the panel members to answer further questions.

Exhibit:

A small exhibit of representative instruments and equipments was planned for continuous showing throughout the day as listed below.

Beta Gages and Radiographic Equipment—Tracerlab, Inc.

Complete Display of Radiation Detectors, Geiger, Proportional, Scintillation Ion Chember for Liquids, Solids, and Gases—Radiation Counter Laboratories, Inc.

Scintillation Counter, Radiation Monitor, and Scintillation Portable Probe—General Electric Co.

One Laboratory Type Radioisotope Analyzer System—Nuclear Instrument and Chemical Corp.

THIRTEENTH SESSION—SYMPOSIUM ON TECHNIQUES FOR ELECTRON METALLOGRAPHY[4]

TUESDAY, JUNE 30, 2:30 P.M.

SESSION CO-CHAIRMEN: C. M. SCHWARTZ AND L. L. WYMAN

Introduction—C. M. Schwartz.

Techniques Used in Electron Microscopy of Aluminum Alloys—M. S. Hunter and F. Keller, presented from manuscript by Mr. Keller.

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[4] Issued as separate technical publication *ASTM STP No. 155.*

Techniques for the Study of Precipitated Carbides—W. D. Forgeng and John L. Lamont, presented by Mr. Forgeng.

Inorganic Replication: Interpretation of Electron Micrographs—C. J. Calbick, presented from manuscript by the author.

Electron Microstructure of Steel by "Extraction Replica" Technique—R. M. Fisher, presented from manuscript by the author.

(*Symposium Continued in Seventeenth Session*)

FOURTEENTH SESSION—STEEL

TUESDAY, JUNE 30, 2:30 P.M.

SESSION CO-CHAIRMEN: VICE-PRESIDENT N. L. MOCHEL AND E. F. LUNDEEN

Papers:

Properties and Behavior of a High-Strength Rivet Steel—Frank Baron and Edward W. Larson, Jr., presented by Mr. Larson.

The Influence of Strain Rate and Temperature on the Strength and Ductility of Mild Steel in Torsion—C. E. Work and T. J. Dolan, presented by Mr. Dolan.

Creep and Creep-Rupture of Some Ferritic Steels Containing 5% to 17% Chromium—E. J. Dulis and G. V. Smith, presented by Mr. Smith.

Influence of Sharp Notches on the Stress-Rupture Characteristics of Heat-Resisting Alloys: Part II—W. F. Brown, Jr., M. H. Jones, and D. P. Newman, presented by Mr. Brown.

Time-Temperature Dependence of the Notch Effect and Influence of Notch Depth in Stress-Rupture Tests on a Cr-Mo-V Steel—D. P. Newman, M. H. Jones, and W. F. Brown, Jr., presented by Mr Brown.

An Investigation of the Time-Temperature Relations for Correlation and Extrapolation of Stress-Rupture Data—S. S. Manson and W. F. Brown, Jr., presented from manuscript by Mr. Manson.

(*Papers on Steel Continued in Thirty-second Session*)

FIFTEENTH SESSION—GILLETT MEMORIAL LECTURE

TUESDAY, JUNE 30, 4:45 P.M.

SESSION CHAIRMAN: PRESIDENT H. L. MAXWELL

Second Gillett Memorial Lecture:

President Maxwell called upon Mr. H. C. Cross of Battelle Memorial Institute, who remarked upon the establishment of the H. W. Gillett Memorial Lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Institute to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first Director of Battelle Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

Mr. Cross introduced Jerome Strauss, Vice-President, Vanadium Corporation of America, who presented the second H. W. Gillett Memorial Lecture on the subject "Micrometallurgy." Mr. Strauss' lecture involved the significance and effect of very small—in some cases minute—quantities of elements on various metals and alloys. Fractions of a per cent of some elements have an important bearing on the use of alloys—frequently beneficial, occasionally deleterious. Small amounts of boron in steel, for example, have important effects in improving the heat-treating properties. Nitrogen and other elements influence the properties. Mr. Strauss covered many other similar situations.

President Maxwell expressed appreciation to Mr. Strauss for his splendid lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

SIXTEENTH SESSION—FATIGUE

TUESDAY, JUNE 30, 8:00 P.M.

SESSION CO-CHAIRMEN: W. T. LANKFORD AND R. R. MOORE

Committee E-9 on Fatigue:

Report presented by R. E. Peterson, chairman, and accepted as a report of progress.

Papers:

The Effect of Range of Stress on Fatigue Strength of Notched and Unnotched SAE 4340 Steel in Bending and Torsion—W. N. Findley, F. C. Mergen, and A. H. Rosenberg, presented from manuscript by Mr. Findley.

Influence of Grain Size on Fatigue Notch-Sensitivity—R. W. Karry, and T. J. Dolan, presented by Mr. Karry.

Static and Fatigue Properties of Carbon, Silicon, and High-Strength Low-Alloy Steel Plates Having a Hole—Frank Baron and Edward W Larson, Jr., presented by Mr. Baron.

Strength Properties of Rolled Aluminum Alloys Under Various Combinations of Alternating and Mean Axial Fatigue Stresses—B. J. Lazan and A. A. Blatherwick, presented by Mr. Lazan.

(*Papers on Fatigue Continued in Twenty-first Session*)

SEVENTEENTH SESSION—SYMPOSIUM ON TECHNIQUES FOR ELECTRON METALLOGRAPHY[5]

(*Continued from Thirteenth Session*)

TUESDAY, JUNE 30, 8:00 P.M.

SESSION CO-CHAIRMEN: G. E. PELLISSIER AND L. L. WYMAN

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and accepted as a report of progress.

Appended Paper:

Techniques for Ferrous Electron Metallography I. Silica and Plastic Replicas. II. Etchants and Etch Depth—W. L. Grube, presented by the author.

Symposium Papers:

Metal-Shadowing for Contrast Enhancement—Comparison of Shadow Metal and Shadow Angle—D. M. Teague, presented from manuscript by the author.

Specimen Polishing Techniques for Electron Metallography of Steel—W. L. Grube and S. R. Rouze, presented from manuscript by Mr. Rouze.

Techniques Which Permit Successive Examinations of Specific Areas by Electron Microscopy—F. A. McLauchlan, presented from manuscript by the author.

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[5] Issued as separate technical publication *ASTM STP No. 155.*

Replica Washing Methods—E. F. Fullam, presented by the author.

Determination of Surface Properties for Eutectoid Steel and Iron as Prepared by the Conventional Metallographic Techniques of Abrading, Mechanical Polishing, and Chemical Etching—C. F. Tufts, presented from manuscript by the author.

Application of New Etchants for Delineation of Fine Structure in Steel—Alfred E. Austin, presented by the author.

Positive versus Negative Plastic Replicas—C. M. Schwartz, presented by the author.

EIGHTEENTH SESSION—ON SIGNIFICANCE OF TESTS OF CONCRETE

TUESDAY, JUNE 30, 8:00 P.M.

SESSION CHAIRMAN: A. T. GOLDBECK

Papers:

Porosity Determinations and the Significance of Pore Characteristics of Aggregates—D. W. Lewis, W. L. Dolch, and K. B. Woods, presented by Mr. Dolch.

Significance of Tests for Chemical Reactions of Aggregates in Concrete—William Lerch, presented by the author.

Significance of Tests on Sulfate Resistance of Concrete—Elmo C. Higginson and Omar J. Glantz, presented from manuscript by Mr. Higginson.

Wear Tests of Concrete—H. L. Kennedy and M. E. Prior, presented from manuscript by Mr. Kennedy.

Pulse Velocity Testing of Concrete—W. E. Parker, presented from manuscript by T. G. Clendenning.

Evaluation of Curing Compounds for Portland Cement Concrete—C. E. Proudley, presented from manuscript by the author.

NINETEENTH SESSION—ATMOSPHERIC SAMPLING, WATER, AND SIMULATED TESTING

WEDNESDAY, JULY 1, 9:00 A.M.

SESSION CHAIRMAN: L. C. MCCABE

Committee D-22 on Methods of Atmospheric Sampling and Analysis:

Report presented by L. C. McCabe, chairman, and accepted as a report of progress.

Papers:

Properties of Various Filtering Media for Atmospheric Dust Sampling—Walter J. Smith and Norman F. Suprenant, presented from manuscript by Mr. Smith.

Some Instrumentaton and Analytical Techniques for the Continuous Evaluation of Air Contaminants—Morris Katz and George D. Clayton, presented from manuscript by Mr. Katz.

Variables in Monthly Dust Fall Measurements—J. Deane Sensenbaugh and W. C. L. Hemeon, presented by George F. Haines.

A Comparison of Methods of Testing for Dissolved Oxygen in Waters of High Purity—Walter L. Riedel presented from manuscript by the author.

Philosophy of Simulated Service Testing—S. A. Gordon, presented from manuscript by the author.[6]

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[6] Published in the ASTM BULLETIN, No. 193, October, 1953, p. 27 (TP 159).

TWENTIETH SESSION—SYMPOSIUM ON LATERAL PILE LOAD TESTS[7]

WEDNESDAY, JULY 1, 9:00 A.M.

SESSION CHAIRMAN: A. E. CUMMINGS

Introduction—A. E. Cummings.

Resistance of Long Hollow Piles to Applied Lateral Loads—G. A. McCammon and J. C. Ascherman, presented by Mr. Ascherman.

Lateral Load Tests on Groups of Battered and Vertical Piles—Lawrence B. Feagin, presented by the author.

Bearing Piles Subjected to Horizontal Loads—L. T. Evans, presented from manuscript by Mr. F. J. Converse.

The Resistance to Lateral Loading of Single Piles and of Pile Groups—G. P. Tschebotarioff, presented by the author.

(Symposium Continued in Twenty-third Session)

PAPERS ON BITUMINOUS WATERPROOFING AND ROOFING MATERIALS[8]

WEDNESDAY, JULY 1, 9:00 A.M.

At a special technical papers session of the meeting of Committee D-8 on Bituminous Waterproofing and Roofing Materials, the following three papers were presented:

Preparation of Bituminous Films by Spinning—Lawrence R. Kleinschmidt.

A Method of Preparing Uniform Films of Bituminous Films—Sidney H. Greenfeld.

Effects of Thermal Shock on the Durability of Asphalt Coatings Under Accelerated Test—Sidney H. Greenfeld.

TWENTY-FIRST SESSION—FATIGUE

(Continued from Sixteenth Session)

WEDNESDAY, JULY 1, 9:00 A.M.

SESSION CO-CHAIRMEN: H. R. NEIFERT AND R. E. PETERSON

Papers:

An Investigation of the Effects of Overstress on the Fatigue Characteristics of Certain Wrought Sheet Magnesium Alloys—Joseph Viglione and Forrest S. Williams, presented from manuscript by Mr. Williams.

Investigation of Prot Accelerated Fatigue Test—E. J. Ward, D. C. Schwartz, and R. T. Schwartz, presented from manuscript by Mr. Ward.

Elevated Temperature Fatigue Properties of SAE 4340 Steel—W. J. Trapp and R. T Swartz, presented from manuscript by E. J. Ward.

Damping, Elasticity, and Fatigue Properties of Unnotched and Notched N-155 at Room and Elevated Temperatures—L. J. Demer and B. J. Lazan, presented from manuscript by Mr. Lazan.

A Random Fatigue Testing Procedure and Machine—A. M. Freudenthal, presented from manuscript by the author.

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[7] Issued as separate technical publication *ASTM STP No. 154.*

[8] Published in the ASTM BULLETIN, No. 193, October, 1953, p. 46 (TP 178).

TWENTY-SECOND SESSION—REPORT SESSION

WEDNESDAY, JULY 1, 11:30 A.M.

SESSION CHAIRMAN: R. C. ADAMS

Committee D-16 on Industrial Aromatic Hydrocarbons:

Reported presented from manuscript, in the absence of the chairman, by M. D. Huber, and accepted as a report of progress.

Committee D-19 on Industrial Water:

Report presented, in the absence of the chairman, by M. D. Huber, and the following actions taken:

Accepted as Tentative:

Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water (D 1252 - 53 T)
Test for Residual Chlorine in Industrial Water (D 1253 - 53 T)
Test for Nitrite Ion in Industrial Water (D 1254 - 53 T)
Test for Sulfides in Industrial Waste Water (D 1255 - 53 T)
Scheme for Analysis of Industrial Water (D 1256 - 53 T)

Accepted as Tentative, Revisions in:

Test for Hardness in Industrial Water (D 1126 - 50 T)

Adopted as Standard, Revisions in:

Definitions of Terms Relating to Industrial Water (D 1129 - 51)

Accepted for Publication as Information Only:

Test for Odor of Industrial Waste Water
Method for Determination of pH of Industrial Waste Water
Test for Residue on Evaporation of Filtered Industrial Waste Water

Committee E-11 on Quality Control of Materials:

Report presented, in the absence of the chairman, by M. D. Huber, and accepted as a report of progress.

Committee E-12 on Appearance:

Report presented, in the absence of the chairman, by M. D. Huber, and the following action taken:

Accepted as Tentative:

Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (E 98 - 53 T) (This test has been developed in cooperation with Committees C-22, D-1, and D-6.)

Committee E-13 on Absorption Spectroscopy:

Report presented by E. J. Rosenbaum, chairman, and accepted as a report of progress.

Committee E-14 on Mass Spectrometry:

Report presented, in the absence of the chairman, by M. D. Huber, and accepted as a report of progress.

TWENTY-THIRD SESSION—SYMPOSIUM ON LATERAL PILE LOAD TESTS

(*Continued from Twentieth Session*)

WEDNESDAY, JULY 1, 2:00 P.M.

SESSION CHAIRMAN: L. E. GREGG

The Lateral Load Capacity of Timber Pile Groups—J. O'Halloran, presented from manuscript by R. F. Legget.
Lateral Load Tests on Vertical Fixed-Head and Free-Head Piles—S. M. Gleser, presented by the author.
Lateral Load Tests on Piles for Design Information—A. A. Wagner, presented from manuscipt by the author.
Summary—A. E. Cummings.

TWENTY-FOURTH SESSION—CONCRETE AND REPORT SESSION

WEDNESDAY, JULY 1, 2:00 P.M.

SESSION CHAIRMAN: STANTON WALKER

Papers:

Some Effects of the Grading of Sand on Masonry Mortar—C. C. Connor, presented by the author.

Thermal Expansion Tests on Aggregates, Neat Cements and Concretes—Leonard J. Mitchell, presented by H. S. Meissner.

Prediction of Concrete Durability from Thermal Tests of Aggregate—Elmo C. Higginson and Donald G. Kretsinger, presented from manuscript by Mr. Higginson.

Determination of Compressive Strength of Concrete by Using Sonic Properties—Clyde E. Kesler and Yoshiro Higuchi, presented by Mr. Kesler.

Comparison of Dynamic Methods of Testing Concretes Subjected to Freezing and Thawing—G. M. Batchelder and D. W. Lewis, presented by Mr. Lewis.

An Investigation of Design Methods for Asphaltic Paving Mixtures—B. A. Vallerga, and Ernest Zube, presented from manuscript by Mr. Vallerga.

Committee C-7 on Lime:

Report presented by J. A. Murray, chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 49)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by Fred Hubbard, vice-chairman, in the absence of the chairman, and the following actions taken:

Accepted as Tentative:

Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 53 T)

Test for Fly Ash as an Admixture for Portland-Cement Concrete (C 311 – 53 T)

Spec. for Liquid Membrane-Forming Compounds for Curing Concrete (C 309 – 53 T)

Accepted as Tentative, Revisions in:

Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156 – 52 T)

Test for Bleeding of Concrete (C 232 – 49 T)

Test for Coal and Lignite in Sand (C 123 – 44)

Adopted as Standard:

Spec. for Waterproof Paper for Curing Concrete (C 171 – 49 T)

Adopted as Standard, Revisions in:

Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (C 289 – 46)

Sanford E. Thompson Award

The Eleventh Sanford E. Thompson Award was made to Mrs. Katharine Mather, Chief, Petrography Section, Concrete Research Division, Waterways Experiment Station, Corps of Engineers, U. S. Army, Jackson, Miss., for her paper on "Applications of Light Microscopy in Concrete Research," presented at the 1952 Annual Meeting and published in the Symposium on Light Microscopy.

Committee C-11 on Gypsum:

Report presented by H. F. Gardner, vice-chairman, in the absence of the chairman, and accepted as a report of progress.

Committee C-12 on Mortars for Unit Masonry:

Report presented by J. M. Hardesty, chairman, and accepted as a report of progress.

Committee D-1 on Paint, Varnish, Lacquer and Related Products:

Report presented by W. T. Pearce, chairman. The report as preprinted indicated revisions of 5 standards for immediate adoption: namely, D 523 – 51, D 605 – 42, D 154 – 50, D 333 – 40 and D 268 – 49. In making his presentation, Dr. Pearce reported that this was in error and that the revision of only one standard was being recommended for immediate adoption: namely, D 268 – 49. The revisions of the other 4 standards were recommended for publication as tentative. The following is a record of the actions taken:

Accepted as Tentative:

Spec. for High-Gravity Glycerine (D 1257 – 53 T)

Methods of Sampling and Testing High-Gravity Glycerine (D 1258 – 53 T)

Test for Nonvolatile Content of Resin Solutions (D 1259 – 53 T)

Method for Calculating Small Color Differences from Data Obtained on the Hunter Multipurpose Reflectometer (D 1260 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Dehydrated Castor Oil (D 961 – 51 T)
Methods of Testing Varnishes (D 154 – 52 T)
Test for 60-deg Specular Gloss (D 523 – 51)
Spec. for Magnesium Silicate Pigment (D 605 – 42)
Methods of Testing Varnishes (D 154 – 50)
Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 40)

Adopted as Standard:

Methods of Testing Drying Oils (D 555 – 51 T)
Test for Roundness of Glass Spheres (D 1155 – 51 T)
Test for Heptane Number of Hydrocarbon Solvents (D 1132 – 50 T)
Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents (D 1134 – 50 T)
Methods of Testing Varnishes (D 154 – 52 T) (Sections 3 to 20 inclusive, covering Drying Time Test and Rosin-Pentaerythrital Ester Flexibility Test). Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer and Related Products on Test Panels (D 823 – 51 T)
Method for Measurement of Dry Film Thickness of Nonmagnetic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Magnetic Base (D 1186 – 51 T)

Adopted as Standard, Revisions in:

Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 49)

Withdrawal of Standard:

Spec. for Perilla Oil, Raw or Refined (D 125 – 48)

Accepted for Publication as Information only:

Test for Viscosity Reduction Power of Hydrocarbon Solvents
Test for Flash Point by the Tag Open-Cup

Committee D-6 on Paper and Paper Products:

Report presented by W. R. Willets, chairman, and the following actions taken:

Accepted as Tentative:

Test for Dimensional Changes of Paper with Changes in Moisture Conditions (D 1270 – 53 T)

Accepted as Tentative, Revisions in:

Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (D 644 – 44)

Adopted as Standard:

Test for Water-Soluble Matter in Paper (D 1162 – 51 T)
Test for Lint of Paper Towels (D 1163 – 51 T)
Test for Ring Crush of Paperboard (D 1164 – 51 T)

The committee withdrew from the report as preprinted the recommendation for adoption as standard of the Tentative Methods of Test for Water Vapor Permeability of Paper and Paperboard (D 988 – 51 T).

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Asphalt Roofing Surfaced with Mineral Granules (D 249 – 50 T)
Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 – 52)
Spec. for Wide Selvage Asphalt Roofing Surfaced with Mineral Granules (D 371 – 51)

TWENTY-FIFTH SESSION—MARBURG LECTURE, DUDLEY MEDAL, AND AWARDS

WEDNESDAY, JULY 1, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT H. L. MAXWELL
CO-CHAIRMAN: VICE-PRESIDENT L. C. BEARD

Edgar Marburg Lecture:

President Maxwell presented Dr. Beard, the newly elected President of the Society, who introduced Dr. Frederick D. Rossini as the Twenty-seventh Marburg Lecturer. He made mention of his many accomplishments in the field of thermo-chemistry, chemical thermodynamics, hydrocarbons, and petroleum. Dr. Rossini, who is Silliman Professor and Head of the Department of Chemistry and Director of the American Petroleum Institute Research Laboratory at the Carnegie Institute of Technology presented a lecture entitled "An Excursion in Petroleum Chemistry." Outstanding developments in our knowledge of petroleum were described, and the interesting story of fundamental research in petroleum chemistry as performed in the laboratories of petroleum companies was outlined. Dr. Rossini discussed projects supported cooperatively by the petroleum industry through the American Petroleum Institute, and in particular reviewed API Research Projects covering the composition of crude petroleum and the researches conducted to make available to the laboratories of the petroleum industry and to the technical work all of the known data on hydrocarbons and related compounds. This extensive laboratory work involved searching the entire scientific literature appraising and arranging data in a useful form, and distributing these data both on a national and international scale.

President Maxwell expressed appreciation to Dr. Rossini for his very interesting lecture on a most important subject and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

Charles B. Dudley Medal:

The Twenty-fifth Award of the Charles B. Dudley Medal was made to E. A. Davis and M. J. Manjoine, Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa., for their paper on "Effect of Notch Geometry on Rupture Strength at Elevated Temperatures," presented at the 1952 Annual Meeting and published in the Symposium on Effects of Notches and Metallurgical Changes on Strength and Ductility of Metals at Elevated Temperatures.

Richard L. Templin Award:

The Eighth Richard L. Templin Award was made to W. N. Findley, Research Associate Professor, and P. G. Jones, Associate Professor, Department of Theoretical and Applied Mechanics, University of Illinois; W. I. Mitchell, Assistant Professor of Civil Engineering, South Dakota School of Mines; and R. L. Sutherland, Assistant Professor of Mechanical Engineering, University of Iowa, for their paper on "Fatigue Machines for Low Temperatures and for Miniature Specimens," presented at the 1952 Annual Meeting and published in ASTM BULLETIN, No. 184, September, 1952.

Sam Tour Award:

The Fourth Sam Tour Award was made to J. R. McDowell, Mechanics Division, Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa., for his paper on "Fretting Corrosion Tendencies of Several Combinations of Materials," presented at the 1952 Annual Meeting and published in the Symposium on Fretting Corrosion.

TWENTY-SIXTH SESSION—SYMPOSIUM ON PORCELAIN ENAMELS AND CERAMIC COATINGS AS ENGINEERING MATERIALS[9]

THURSDAY, JULY 2, 9:00 A.M.

SESSION CHAIRMAN: DWIGHT G. BENNETT

Committee C-22 on Porcelain Enamel:

Report presented by W. N. Harrison, chairman, and the following actions taken:

Accepted as Tentative:

Test for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal (C 313 – 53 T)

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[9] Issued as separate technical publication *ASTM STP No. 153.*

Test for Warpage of Porcelain Enameled Flatware (C 314 – 53 T)

Adopted as Standard:

Test for Acid Resistance of Porcelain Enamels (Room Temperature Test) (C 282 - 51 T)

Symposium Papers:

Introduction—Dwight G. Bennett.

Some Examples of the Functional Use of Porcelain Enamel and Ceramic Coatings—G. H. Spencer-Strong, presented from manuscript by the author.

Resistance of Porcelain Enamels to Weathering —Dwight G. Moore, presented by the author.

The Chemical Resistance of Glass Fused to Steel —O. J. Britton and E. A. Sanford, presented by Mr. Britton.

Acid Resisting Properties of Porcelain Enamels —Harold C. Wilson, presented by the author.

Requirements for and Expected Benefits from the Application of Coatings to High Temperature Components of a Jet Engine—Allen C. Francisco and G. M. Ault, presented from manuscript by Mr. Ault.

High Temperature Ceramic Coatings as Applied to Aircraft Power Plants—B. L. Paris, presented by D. G. Bennett.

The Industrial Processing of High Temperature Ceramic Coatings—J. H. Terry, presented from manuscript by the author.

A Laboratory Evaluation of Ceramic Coatings for High Temperature Applications—Sara J. Ketcham, presented by the author.

(*Symposium Continued in Twenty-eighth Session*)

TWENTY-SEVENTH SESSION—PLASTICS

THURSDAY, JULY 2, 2:00 P.M.

SESSION CHAIRMAN: G. M. KLINE

Committee D-20 on Plastics:

Report presented by R. K. Witt, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Nylon Injection Molding and Extrusion Compounds (D 789 – 44 T)

Definitions of Terms Relating to Plastics (D 883 – 52 T)

Spec. for Vinyl Chloride-Acetate Resin Plastic Sheets (D 708 – 50)

Adopted as Standard:

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 51 T), with the addition of the following sentence to Note 2 in Section 4: "Shorter conditioning times may be used for thin specimens provided equilibrium is substantially obtained." (Jointly with Committee D-9 on Electrical Insulating Materials)

Committee D-9 on Electrical Insulating Materials:

Report presented by A. H. Scott, chairman, and the following actions taken:

Accepted as Tentative:

Test for Corrosive Sulfur in Electrical Insulating Oils (D 1275 – 53 T)

Accepted as Tentative, Revisions in:

Methods of Testing Pressure-Sensitive Adhesive Tapes Used for Electrical Insulation (D 1000 – 48 T)

Spec. for Natural Muscovite Mica Based on Visual Quality (D 351 – 52 T): with the following further revisions:

Section 4 (*a*).—Include an additional definition: "Crystallographic Discloloration appears as bands of lighter or darker shades of the basic color of the block of mica. Such bands generally are parallel to the crystallographic faces of the crystal from which the block was separated"

Section 5 (*a*) —Substitute the following new description for the procedure for grading full trimmed mica.

"For full trimmed mica, all mica blocks, thins, and condenser films shall be fully trimmed to remove all cracks, holes, reeves and crossgrains according to the quality desired. Trimming should follow the natural contour of mica. As far as possible, all marginal cracks should be removed by recutting. The total area of each piece for Grade 4 and larger shall not be more than 1.54 times the area of the largest usable rectangle or rectangular yield of 65 per cent.

"For grades 5 and smaller the total area of each piece shall not be more than 2 times the area of the largest usable rectangle or rectangular yield of 50 per cent.

"The usable area is the area of maximum rectangle obtainable.

"If limitation as to the size, number and frequency of 'V' cuts is desired, it shall be subject to agreement between purchaser and vendor."

Table I.—Change the dimension of one size for Grade A-1 (Special) from "4" to "3½ in."

Table II.—Add a new footnote *h* to the heading "Clear Uniform Color" to read as follows:

"Crystallographic discoloration is permitted to a limited extent in V-4 Good Stained quality. It is not permitted in qualities higher than V-4 Good Stained. In qualities below V-4 Good Stained it is permitted without limit."

Add an additional classification "V-11, Densely Stained," and provide check marks under all columns except columns headed "Black Stains (Mineral) *g*" and "Red Stains (Mineral) *e*" under which place *X* marks.

Section 9 (a).—Add a new visual quality designation to the Table as follows:

ASTM Visual Quality	*Commercial Designation*
V-11	Densely Stained

Appendix I.—Add the following verbal description of visual quality: "V-11, Densely Stained.—Soft and may contain all heavy stains and inclusions and other defects, heavy waves, cracks and buckles."

Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation (D 202 – 52 T)

Spec. for Flexible Treated Cotton and Rayon Sleeving Used in Electrical Insulation (D 372 – 47 T)

Methods of Testing Electrical Insulating Oils (D 117 – 50 T)

Methods of Testing Askarels (D 901 – 52 T)

Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 48)

Adopted as Standard:

Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 52 T), as revised. The report as preprinted included a revision of Section 27 by the inclusion of a new Paragraph (*f*). This paragraph was withdrawn by the committee and a revision of Section 28 was proposed instead to read as follows:

28. The report shall include the following:

(*1*) The test method used for determining the dielectric strength,

(*2*) The average, maximum and minimum dielectric breakdowns in volts for each test method,

(*3*) The average, maximum and minimum thicknesses of the sample as determined in Section 11, and

(*4*) The average dielectric strength in volts per mil obtained by dividing the average dielectric breakdown in (*2*) by the average thickness in (*3*).

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 51 T) with addition of a sentence in Note under Section 4 as indicated under Committee D-20 on Plastics.

Papers:

Determination of Elastic Constants of Orthotropic Materials with Special Reference to Laminates—R. K. Witt, W. H. Hoppmann II, and R. S. Buxbaum, presented by Mr. Hoppmann.[10]

Three Years Outdoor Weather Aging of Plastics Under Various Climatological Conditions—S. E. Yustein, R. R. Winans, and H. J. Stark, presented by Mr. Yustein.[11]

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[10] Published in the ASTM BULLETIN, No. 194, December, 1953, p. 53 (TP 197).

[11] Published in ASTM BULLETIN, No. 196, February, 1954, p. 29 (TP 37).

TWENTY-EIGHTH SESSION—SYMPOSIUM ON PORCELAIN ENAMELS AND CERAMIC COATINGS AS ENGINEERING MATERIALS

(*Continued from Twenty-sixth Session*)

THURSDAY, JULY 2, 2:30 P.M.

SESSION CHAIRMAN: W. N. HARRISON

Abrasion Resistance of Various Types of Porcelain Enamel—A. V. Sharon, presented from manuscript by the author.

The Resistance of Porcelain Enamels to Surface Abrasion as Determined by the PEI Test.—John T. Roberts, presented from manuscript by the author.

Torsion Testing as an Aid to the Porcelain Enamel Industry—Earl Hoover, presented from manuscript by the author.

The Strengthening Effect of Porcelain Enamel on Sheet Iron as Indicated by Bending Tests—E. E. Bryant, presented from manuscript by the author.

Tension Tests of Porcelain Enameled Steels—W. A. Deringer, presented by the author.

The Effect of Temperature on the Electrical Resistivity of Several Silicone and Ceramic-Type Coatings—Simon W. Strauss, Lloyd E. Richards, and D. G. Moore, presented from manuscript by Mr. Strauss.

In Retrospect and in Prospect—Summary—W. N. Harrison.

TWENTY-NINTH SESSION—SYMPOSIUM ON DYNAMIC TESTING OF SOILS[12]

THURSDAY, JULY 2, 2:30 P.M.

SESSION CHAIRMAN: F. J. CONVERSE

Introduction—R. K. Bernhard.

Elasticity and Damping of Oscillating Bodies on the Soil—Ing. H. Lorenz, presented from manuscript by R. K. Bernhard.

The Pressures Generated in Soil by Compaction Equipment—A. C. Whiffin, presented by R. K. Bernhard.

Loose Sands—Their Compaction by Vibroflotation—Elio D'Appolonia, presented from manuscript by the author.

Performance Records of Engine Foundations—Gregory P. Tschebotarioff, presented by the author.

Makromeritic Liquids—Hans F. Winterkorn, presented from manuscript by the author.

(*Symposium Coninued in Thirtieth Session*)

THIRTIETH SESSION—SYMPOSIUM ON DYNAMIC TESTING OF SOILS[12]

(*Continued from Twenty-ninth Session*)

THURSDAY, JULY 2, 8:00 P.M.

SESSION CHAIRMAN: R. K. BERNHARD

Pilot Studies on Soil Dynamics—R. K. Bernhard and J. Finelli, presented from manuscript by Mr. Finelli.

Compaction of Sand at Resonant Frequency—F. J. Converse, presented by the author.

The Elastic Theory of Soil Dynamics—Patrick M. Quinlan, presented from manuscript by the author.

A Dynamic Analogy for Foundation-Soil Systems—Adrian Pauw, presented from manuscript by the author.

A Discontinuous Model for the Problems of Soil Dynamics—J. J. Slade, Jr., presented from manuscript by the author.

Vibrations in Semi-Infinite Solids Due to Periodic Surface Loading—Tse Yung Sung, presented from manuscript by the author.

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[12] Issued as separate technical publication *ASTM STP No. 156*.

THIRTY-FIRST SESSION—CORROSION

Thursday, July 2, 8:00 p.m.

Session Chairman: I. V. Williams

Advisory Committee on Corrosion:

Report presented by J. S. Pettibone, secretary, in the absence of the chairman, and accepted as a report of progress.

Committee A-5 on Corrosion of Iron and Steel:

Report presented by A. P. Jahn, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (A 93 – 52 T)
Spec. for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 – 50 T)

Adopted as Standard, Revisions in:

Test for Weight of Coating on Zinc-Coated Iron or Steel Articles (A 90 – 39)
Spec. for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars, and Their Products (A 123 – 47)
Spec. for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 49)

Withdrawal of Tentative:

Spec. for Lead-Alloy Coating on Iron or Steel Hardware (A 267 – 46 T)

Appended Paper:

Some Observations on the Preece Test and Stripping Tests for Zinc Coated Wires—G. A. Ellinger, T. H. Orem, and W. J. Pauli, presented by Mr. Ellinger.

Committee A-9 on Ferro-Alloys:

Report presented, in the absence of the chairman, by H. B. Myers, and the following action taken:

Accepted as Tentative, Revisions in:

Spec. for Ferromanganese (A 99 – 50)

The standard was reverted to tentative in its revised form.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented, in the absence of the chairman, by J. S. Pettibone, and the following actions taken.

Accepted as Tentative:

Spec. for Stainless Steel Wire Strand (A 368 – 53 T)

Adopted as Standards, Revision in:

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 47) (Jointly with Committee A-1 on Steel)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 47): Covering (1) adoption as standard of present-tentative revision of Section 12(*a*); and (2) immediate adoption of revision of Table II. (Jointly with Committee A-1 on Steel)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by A. W. Tracy, secretary, in the absence of the chairman, and accepted as a report of progress.

Appendix:

Progress Report of Sub-Group of Committee B-3, Subcommittee VII, on corrosiveness of Various Atmospheric Test Sites as Measured by Specimens of Steel and of Zinc—C. P. Larrabee and O. B. Ellis, presented from manuscript by Mr. Larrabee.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman. The report included a recommendation for the immediate revision of Standard Specifications for Zinc-Base Alloy Die Castings (B 86 – 52) which would provide for an increased permissible copper content in Alloy XXIII. Since this proposed revision had not been published previously as tentative, it required a nine-tenths vote for approval. The recommendation failed to receive the necessary nine-tenths vote.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman. and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 51 T)

Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 49 T)

Spec. for Aluminum and Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 - 52 T)

Spec. for Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 52 T)

Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 - 52 T)

Spec. for Aluminum and Aluminum-Alloy Bars, Rods and Wire (B 211 - 52 T)

Spec. for Magnesium-Base Alloy Extruded Round Tubes (B 217 - 49 T)

Spec. for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 52 T)

Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat exchangers (B 234 - 50 T)

Spec. for Aluminum-Alloy Extruded Tubes (B 235 - 52 T)

Spec. for Aluminum-Alloy Pipe (B 241 - 50 T)

Spec. for Aluminum and Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Applications (B 273 - 52 T)

Spec. for Aluminum and Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 - 52 T)

Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 52 T)

The committee withdrew from the report as preprinted the recommendation for the revision in Table I of the chromium range for alloy GS11A in the following specifications: B 178, B 209, B 210, B 211, B 221, B 235, B 241, B 273 and B 274.

Adopted as Standard:

Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 51 T)

Committee E-1 on Methods of Testing:

Report presented by P. J. Smith, secretary, in the absence of the chairman, and the following actions taken:

Accepted as Tentative:

Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 - 53 T)

Accepted as Tentative, Revisions in:

Spec. for ASTM Thermometers (E 1 - 52) (Addition of three Kinematic Viscosity Thermometers, 72F, 73F, and 74F)

Adopted as Standard:

Spec. for ASTM Thermometers (E 1 - 52) (The 18 specifications for ASTM Precision Thermometers 62C to 70C and 62F to 70F now published as tentative in Table I of Specifications E 1.)

Adopted as Standard, Revisions in:

Spec. for ASTM Thermometers (E 1 - 52) for immediate adoption

Committee E-7 on Non-Destructive Testing:

Report presented by J. H. Bly, chairman, and the following actions taken:

Accepted as Tentative:

Reference Radiographs of Aluminum and Magnesium Castings (E 98 - 53 T)

Papers:

The Corrosion of Beryllium Copper Strip in Sea Water and Marine Atmospheres—J. T. Richards, presented by the author.

Appendix to B-7 Report:

Resistance of Light Metals to Atmospheric Exposure—L. H. Adam, presented by the author.

THIRTY-SECOND SESSION—STEEL

(*Continued from Fourteenth Session*)

FRIDAY, JULY 3, 9:00 A.M.

SESSION CHAIRMAN: J. R. TRIMBLE

Papers:

Tension Impact Testing of Sheet Metals—Carl W. Muhlenbruch, presented from manuscript by the author.[13]

The Measurement of Directional Strength in Straight and Cross-Rolled Strip by the Navy Tear Test—Helen Hoover, presented by the author.

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[13] Published in ASTM BULLETIN, No. 196, February, 1954, p. 43 (TP 51).

A Comparison of Impact Testing Machines in the 20–30 ft-lb Range—R. L. Rickett, W. B. Seens, R. Roeloffs, R. W. Vanderbeck, and C. Daniel, presented from manuscript by Mr. Rickett.

Delayed Yielding in Annealed Low-Carbon Steel Under Compression Impact—J. E. Johnson, D. S. Wood, and D. S. Clark, presented by by Mr. Johnson.

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Methods and Definitions for the Mechanical Testing of Steel Products (A 370 – 53 T)

Spec. for Cold-Rolled Carbon Steel Deep-Drawing Sheet, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 – 53 T)

Spec. for Cold-Rolled Carbon Steel Sheet, Commercial Quality (A 366 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Steel for Bridges and Buildings (A 7 – 52 T)

Spec. for Quenched Carbon-Steel Joint Bars (A 49 – 51 T)

Spec. for Structural Steel for Ships (A 131 – 52 T)

Spec. for Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 52 T)

Spec. for Electric-Resistance Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 51 T)

Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 52 aT)

Spec. for Heat-Treated Carbon and Alloy-Steel Track Bolts and Nuts (A 183 – 51 T)

Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 52 T)

Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 52 T)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 47 T)

Spec. for Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 49 T)

Spec. for Electric Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 51 T)

Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger and Condenser Tubes (A 249 – 52 T)

Spec. for Electric-Resistance-Welded Carbon Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 51 T)

Spec. for Steel Plates for Pressure Vessels for Service at Low-Temperature (A 300 – 52 T)

Spec. for Minimum Requirements for the Deformation of Deformed Steel Bars for Concrete Reinforcement (A 305 – 50 T)

Spec. for Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 52 T)

Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 52 T)

Spec. for Quenched-and-Tempered Alloy-Steel Bolts and Studs with Suitable Nuts (A 354 – 52 T)

Spec. for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 37)

Adopted as Standard:

Spec. for Electric-Fusion (Arc)-Welded Steel Plate Pipe (Sizes 16 in. and over) (A 134 – 51 T)

Spec. for Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and over) (A 139 – 51 T)

Spec. for Open-Hearth Carbon-Steel Rails (A 1 – 49 T)

Adopted as Standard, Revisions in:

Spec. for High-Carbon Steel Joint Bars (A 5 – 48)

Spec. for Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 51)

The committee withdrew from the report as preprinted the revision of Specifications A 194 relating to Section 9 (*a*).

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 47) (Jointly with Committee A 10)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 52) covering (1) adoption as standard of present tentative revision of Section 12 (*a*), and (2) immediate adoption of revision of Table II. (Jointly with Committee A 10).

THIRTY-THIRD SESSION—REPORT SESSION

FRIDAY, JULY 3, 11:30 A.M.

SESSION CO-CHAIRMEN: PAST PRESIDENT K. G. MACKENZIE AND A. E. MILLER

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by P. J. Smith, and the following actions taken:

Accepted as Tentative:

Test for Rinsing Properties of Metal Cleaners (D 1281 - 53 T)
Test for Buffering Action of Metal Cleaners (D 1279 - 53 T)
Test for Total Immersion Corrosion Test for Soak Tank Metal Cleaners (D 1280 - 53 T)
Test for Analysis of Sodium Bicarbonate (D 501 - 53 T)

Accepted as Tentative, Revisions in:

Spec. for Milled Toilet Soap (D 455 - 48)

Adopted as Standard:

Spec. for Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer) (D 1111 - 51 T)
Spec. for Solid Soap for Low-Temperature Washing (Low and Medium Titer) (D 1112 - 51 T)
Test for Foaming Properties of Surface-Active Agents (D 1173 - 51 T)

Accepted for Publication as Information Only:

Method of Aerated Total Immersion Corrosion Test for Metal Cleaners

Committee D-13 on Textile Materials:

Report presented by W. D. Appel, chairman, and the following actions taken:

Accepted as Tentative:

Test for Average Fiber Diameter of Wool Tops by Porous Plug Tester (D 1282 - 53 T)
Test for Alkali Solubility of Wool (D 1283 - 53 T)
Test for Relaxation and Felting Shrinkage in Laundering of Stabilized Knit Wool Fabrics (D 1284 - 53 T)
Testing and Tolerances for Yarns Containing Wool (D 1285 - 53 T)

Accepted as Tentative, Revisions in:

Definitions of Terms Relating to Textile Materials (D 123 - 52 T)
Spec. and Methods of Test for Fineness of Wool Tops (D 472 - 50 T)
Methods of Testing and Tolerances for Rayon Tire Cord (D 885 - 46 T)
Methods of Test for Clean Wool Content of Wool in the Grease (D 584 - 52 T)
Methods of Core Sampling of Wool in Packages for Determination of Percentage of Hard Scoured Wool Content (D 1060 - 49 T)
Recommended Practice for Designation of Yarn Construction (D 1244 - 52 T)
General Methods of Testing Woven Fabrics (D 39 - 49)

Adopted as Standard:

Definitions of Terms Relating to Textile Materials (D 123 - 52 T): Definitions for 21 items as set forth in the preprinted report, now appearing in Tentative D 123 - 52 T, were recommended by the committee for adoption as standard.
Test for Apparent Fluidity of Dispersions of Cellulose Fibers (D 539 - 51 T)
Methods of Testing Bonded Fabrics (D 1117 - 50 T)
Methods of Testing Twine (From Bast and Leaf Fibers) (D 1233 - 52 T)

Adopted as Standard, Revisions in:

Definition of Terms Relating to Textile Materials (D 123 - 52)
Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 - 52)
Methods of Testing Felt (D 461 - 51)
Spec. for Textile Testing Machines (D 76 - 49)

Withdrawal of Standard:

Spec. and Methods of Testing for Osnaburg Cement Sacks (D 205 - 39)

Withdrawal of Tentatives:

Test for Identification of Finishes on Textiles (D 683 - 42 T)
Spec. and Methods of Test for Woolen Yarns (D 403 - 48 T)
Spec. and Methods of Test for Worsted Yarns (D 404 - 48 T)

Committee D-14 on Adhesives:

Report presented by Gerald Reinsmith, chairman, and the following actions taken:

Accepted as Tentative:

Test for Effect of Mold Contamination on Permanence of Adhesive Preparations and Adhesive Bonds (D 1286 – 53 T)

Adopted as Standard:

Test for Blocking Point of Potentially Adhesive Layers (D 1146 – 51 T)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following action taken:

Adopted as Standard:

Methods of Testing Rosin Oils (D 1131 – 50 T)

The committee withdrew from the report as preprinted the proposed Tentative Specifications for Rosin, and proposed Tentative Specifications for Dipentene.

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

Withdrawal of Tentatives:

Spec. for Materials for Stabilized Base Course (D 556 – 40 T) (Jointly with Committee D-4)
Spec. for Materials for Stabilized Surface Course (D 557 – 40 T) (Jointly with Committee D-4)

Committee D-15 on Engine Antifreezes:

Report presented by H. R Wolf, chairman, and the following actions taken:

Accepted as Tentative:

Test for pH of Concentrated Engine Antifreezes (D 1287 – 53 T)

Accepted as Tentative, Revisions in:

Spec. for Hydrometer Thermometer Field Tester for Engine Antifreezes (D 1124 – 50 T)
Test for Ash Content of Concentrated Engine Antifreezes (D 1119 – 50 T)

Adopted as Standard:

Test for Boiling Point of Engine Antifreezes (D 1120 – 51 T)
Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (D 1122 – 50 T)

Committee D-2 on Petroleum Products and Lubricants:

Report presented by W. T. Gunn, secretary, in the absence of the chairman, and the following actions taken:

Accepted as Tentative:

Test for Effect of Grease on Copper (D 1261 – 53 T)
Test for Lead in New and Used Greases (D 1262 – 53 T)
Test for Leakage Tendencies of Automotive Wheel Bearing Grease (D 1263 – 53 T)
Test for Water Washout Characteristics of Lubricating Greases (D 1264 – 53 T)
Method of Sampling Liquefied Petroleum Gases (D 1265 – 53 T)
Test for Sulfur in Petroleum Products and Liquefied Petroleum Gases by the CO_2-O_2 Lamp Method (D 1266 – 53 T)
Test for Unsaturated Light Hydrocarbons (Silver-Mercuric Nitrate Absorption) (D 1268 – 53 T)
Test for Vapor Pressure of Liquefied Petroleum Gases (D 1267 – 53 T)
Method for Polarographic Determination of Tetraethyllead in Gasoline (D 1269 – 53 T), including a new Note 1 following the Scope to read "Note 1.—Another method, D 526 – 53 T, Test for Tetraethyllead in Gasoline, is also available for this purpose. These two methods give equivalent results within their stated precision limits."

Accepted as Tentative, Revisions in:

Method of Sampling Petroleum and Petroleum Products (D 270 – 52 T)
Test for Kinematic Viscosity (D 445 – 52 T)
Test for Tetraethyllead in Gasoline (D 526 – 48 T), including a new Note 1 following the Scope to read "Note 1.—Another method, D 1269 – 53 T, Polarographic Determination of Tetraethyllead in Gasoline, is also available for this purpose. These two methods give equivalent results within their stated precision limits."
Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents (D 611 – 51 T)
Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water (D 665 – 52 T)
Test for Olefins and Aromatics in Petroleum Distillates (D 875 – 46 T)
Test for Oxidation Characteristics of Inhibited Steam-Turbine Oils (D 943 – 47 T)
Test for Neutralization Value (Acid and Base

Numbers) by Color Indicator Titration (D 974 – 52 T)

Classification of Diesel Fuel Oils (D 975 – 52 T)

The committee withdrew revisions relating to Specifications D 975 – 52 T as set forth in the first, second, and fourth paragraphs of the list of changes for Table I as given in the report as preprinted.

Test for Oxygen in Butadiene Vapors (Manganous Hydroxide Method) (D 1021 – 49 T)

Test for Oil Content of Paraffin Wax (D 721 – 51 T)

Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (D 156 – 49)

Special permission was obtained from the Executive Committee of the Board of Directors to add a recommendation for the revision of Tentative Specifications for Aviation Gasoline (D 910 – 52 T) to include a new 108/135 grade of aviation gasoline.[14]

The committee withdrew from the report as preprinted the proposed revisions in Test for Free and Corrosive Sulfur in Petroleum Products (Copper Strip Test) (D 130 – 50 T).

Adopted as Standard:

Test for Butadiene Dimer in Polymerization Grade Butadiene (D 1024 – 49 T)

Test for Carbonyl Content of Butadiene (D 1089 – 50 T)

Test for Water Tolerance of Aircraft Fuels (D 1094 – 50 T)

ASTM-IP Petroleum Measurement Tables (D 1250 – 52 T)

Test for Boiling Point Range of Polymerization Grade Butadiene (D 1088 – 51 T)

The Committee withdrew from the report as preprinted the recommendation for the adoption as standard of Tentative Method of Test for Foaming Characteristics of Crankcase Oils (D 892 – 46 T).

Adopted as Standard, Revisions in:

Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 – 52)

Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 – 44)

Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 – 52)

Test for Distillation of Natural Gasoline (D 216 – 52)

Definition of Terms Relating to Petroleum (D 288 – 52)

Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 – 49)

Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 – 39)

Method for Calculating Viscosity Index (D 567 – 41)

Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 – 44)

Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 – 51)

Withdrawal of Standard:

Method of Test for Existent Gum in Gasoline (Air-Jet Evaporation Method) (D 381 – 50)

Accepted for Publication as Information Only:

Test for Functional Life of Ball Bearing Greases

Test for Chlorine in Lubricating Oil (Sodium Alcoholate Volumetric Method)

Test for Sodium in Residual Fuel Oil by Flame Photometer

Recommended Practices for Applying Precision Data Given in ASTM Methods of Test for Petroleum Products and Lubricants

Weathering Test for Liquefied Petroleum Gases

Test for Water in Petroleum Products and Other Bituminous Material

Methods for Chemical Analysis of Graphite

Committee D-4 on Road and Paving Materials:

Report presented by C. A. Carpenter, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Bituminous Mixing Plant Requirements (D 995 – 51)

Adopted as Standard:

Spec. for Preformed Expansion Joint Filler for Concrete, Bituminous Type (D 994 – 48 T)

Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 – 51 T)

Adopted as Standard, Revisions in:

Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (D 289 – 46) (Jointly with Committee C-9)

Spec. for Granite Block for Pavements (D 59 – 39)

Spec. for Emulsified Asphalt (D 977 – 49)

Spec. for Bituminous Mixing Plant Requirements (D 995 – 51)

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[14] This recommendation, not preprinted, was accepted subject to unanimous affirmative letter ballot of Committee D-2, which ballot has been favorable.

Withdrawal of Tentatives:

Spec. for Materials for Stabilized Base Course (D 556 – 40 T) (Jointly with Committee D-18)
Spec. for Materials for Stabilized Surface Course (D 557 – 40 T) (Jointly with Committee D-18)

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Non-Rigid Thermoplastic Compounds for Automotive and Aeronautical Applications (D 1277 – 53 T)
Methods for Determining Harmful Dirt in Crude Natural Rubber (D 1278 – 53 T)

Accepted as Tentative, Revisions in:

Test for Compression Set of Vulcanized Rubber (D 395 – 52 T)
Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 – 52 T)
Methods of Testing Compressed Asbestos Sheet Packing (D 733 – 52 T)
Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 – 52 T)
Test for Impact Resilience and Penetration of Rubber by the Rebound Pendulum (D 1054 – 49 T)
Spec. and Methods of Test for Latex Foam Rubbers (D 1055 – 52 T)
Spec. and Methods of Test for Sponge and Expanded Cellular Rubber Products (D 1056 – 52 T)
Test for Compressibility and Recovery of Gasket Materials (D 1147 – 51 T)
Methods of Testing Adhesives for Brake Lining and Other Friction Materials (D 1205 – 52 T)

Adopted as Standard, Revisions in:

Method of Air Pressure Heat Test of Vulcanized Rubber (D 454 – 52)
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 52)
Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 52)

THIRTY-FOURTH SESSION—REPORT SESSION

FRIDAY, JULY 3, 11:30 A.M.

SESSION CO-CHAIRMEN: C. H. FELLOWS AND D. E. PARSONS

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Adopted as Standard:

Spec. for Magnesium Chloride (C 276 – 51 T)
Spec. for Magnesium Sulfate (C 277 – 51 T)

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (C 133 – 49)
Definitions of Terms Relating to Refractories (C 71 – 51)

Adopted as Standard, Revisions in:

Definitions of Terms Relating to Refractories (C 71 – 51)

Committee C-14 on Glass and Glass Products:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Accepted as Tentative, Revisions in:

Method of Sampling Glass Containers (C 224 – 49 T)

Adopted as Standard, Revisions in:

Methods of Chemical Analysis of Soda-Lime Glass (C 169 – 43)

Committee C-15 on Manufactured Masonry Units:

Report presented by M. H. Allen, secretary, in the absence of the chairman, and accepted as a report of progress.

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following action taken:

Accepted as Tentative:

Recommended Practice for Clearance of Preformed Thermal Pipe Insulation (C 312 – 53 T)

Committee C-17 on Asbestos-Cement Products:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following action taken:

Accepted as Tentative, Revisions in:

Spec. for Flat Asbestos-Cement Sheets (C 220 – 49 T)

Committee C-19 on Structural Sandwich Constructions:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Adopted as Standard:

Test for Density of Core Materials for Structural Sandwich Constructions (C 271 – 51 T)
Test for Water Absorption of Core Materials for Structural Sandwich Constructions (C 272 – 51 T)
Method of Shear Test of Sandwich Constructions in Flatwise Plane (C 273 – 51 T)
Definition of Terms Relating to Structural Sandwich Constructions (C 274 – 51 T)

Committee C-21 on Ceramic Whiteware:

Report presented, in the absence of the chairman, by L. C. Gilbert, and accepted as a report of progress.

Committee E-5 on Fire Tests of Materials and Construction:

Report presented by R. C. Corson, secretary, in the absence of the chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Fire Tests of Building Construction of Materials (E 119 – 50)

Adopted as Standard, Revisions in:

Methods of Fire Tests of Building Construction of Materials (E 119 – 50)

(*a*) Revision for immediate adoption with respect to Section 10 of Methods E 119.
(*b*) Adoption of tentative revision as standard, covering the incorporation of new Sections 27 to 30 relating to an Alternate Test of Protection for Structural Steel Beams and Girders.

Withdrawal of Tentative Revision:

Methods of Fire Tests of Building Construction of Materials (E 119 – 50)

Committee C-1 on Cement:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative:

Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (C 305 – 53 T)

Accepted as Tentative, Revisions in:

Test for Bleeding of Cement Pastes and Mortars (C 243 – 52 T)
Test for Air-Content of Hydraulic Cement Mortar (C 185 – 50 T)

Spec. for Portland Blast-Furnace Slag Cement (C 205 – 51 T)

Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 50)

Spec. for Masonry Cement (C 91 – 51)

Adopted as Standard:

Spec. for Air-Entraining Portland Cement (C 175 – 52 T)

Definitions of Terms Relating to Hydraulic Cement (C 219 – 51 T): relating to the definition of the term Addition.

Adopted as Standard, Revisions in:

Spec. for Masonry Cement (C 91 – 52)

(*a*) Revisions for immediate adoption in Sections 19, 24, 25 and 27 of Specifications C 91.

(*b*) Adoption as standard of tentative revisions with respect to Section 7 of Specifications C 91.

Test for Portland Cement by the Turbidimeter (C 115 – 42)

Test for heat of Hydration of Portland Cement (C 186 – 49)

Test for Autoclave Expansion of Portland Cement (C 151 – 49)

Methods of Chemical Analysis of Portland Cement (C 114 – 47)

Spec. for Portland Cement (C 150 – 52)

(*a*) Revision for immediate adoption relating to Table II.

(*b*) Adoption as standard of tentative revisions relating to the SO_3 content and fineness, issued September, 1951, and January, 1952.

"PEOPLE AND THINGS"

ANNUAL ADDRESS BY THE PRESIDENT

H. L. MAXWELL[1]

JUNE 30, 1953

My first thought in seeing you here today is to tell you how grateful I am for the opportunity you entrusted to me a year ago. This year's tour of duty has been a challenge at this particular time. It was an honor to follow the course surveyed and fortified so well by my 45 predecessors in this office. This is the closing week of my year—a very pleasant year—during which I have met and visited with hundreds of ASTM members, some for the first time. It makes one increasingly aware of the personal, even fraternal, bond that unites us.

The official report of the Society's activities for the year is already in your hands. A further elaboration of the financial and technical achievements would be repetitious and therefore inappropriate.

More appropriately, I want to convey to you some of the confident assurance that characterizes our Board of Directors and the Executive Committee, and shared also by the Headquarters Staff. I can best express, briefly, that feeling of the Society's well-being, now as we are entering into our second half century, by borrowing from William Wordsworth who wrote in his confident summary:

No fears to beat away—
No strifes to heal,
The Past is unsighed for, and the future sure.

During the year now closing I have traveled many miles and have spoken at most of the District meetings and several colleges, invariably on technical subjects. For today, I shall use my temporary prerogative, which I shall forfeit now in a few days, and speak on a new topic, "People and Things" for this, my twilight appearance.

Whatever kind of daily work occupies our time and energy, whether it is in designing chemical plants or whipping up a soufflé, whether it is teaching calculus or riding in a roller chair on the boardwalk; our every action—every day—is concerned directly with either people or things or both. The relative priority we as free moral agents recognize in dealing with people and things determines in large measure what kind of individuals we are. It is safe to say that we are drawn naturally to an associate who respects people and uses things. We are just as naturally repelled by one who respects things and uses people.

The ASTM, as a result of careful and forward looking management, has, over the years, accumulated considerable property. We members have a fine building, an additional building lot, some stocks and bonds, and in fact quite a few valuable things as measured on a cash basis. But, by far, the greater value is represented by the individual people, the men and women, who make up the membership, the committees, and Staff of the Society.

Let me illustrate that point. If by some strange and regrettable coincidence all of the physical assets of our Society, including building and financial reserves, were totally destroyed but all of the Society's membership and Staff left intact and active, we would suffer no more than a temporary inconvenience. We would still be in business. We would still be at the lead of the technical societies, taking all steps to replace the physical facilities.

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[1] Technical adviser, Engineering Dept., E. I. du Pont de Nemours and Co., Inc., Wilmington, Del.

If, on the other hand, our Society's physical assets, including building and land and financial resources, remained unchanged and by some catastrophic misfortune the entire membership and Staff were blotted out, the motivating element which is essential for going ahead would be irretrievably lost and the material assets would be lifeless.

Make these and other comparisons by any standards of value you choose, and without exception you come to the inevitable conclusion that material things have value only to the extent that they, ultimately, satisfy some human need.

In every day of our lives, the superiority of people over things is emphasized in many ways. Take, for instance, modern aviation. Some of you, as I, have been in a modern airplane, poised on the runway on the outermost tip of Newfoundland waiting for a favorable radio release on the weather. And when a description of the weather came over the radio, the pilot, belonging to the only species in the whole of the animal kingdom with ability to reason, called on his judgment and made a decision that literally determined matters of life and death for every passenger. And then—we took off that night and traveled through a vast and dark and freezing emptiness, guided unerringly by instruments that were conceived only in the mind of men. Those modern instruments were built as rugged as a blacksmith's anvil—yet fantastically accurate. They were truly the electronic counterpart of the sensitive finger tips of the man who designed them. These mechanical eyes, seen yet unseeing in the usual sense, guided the great plane through the stillness of the Arctic night. Crossing first over the dark green patchwork of rural Ireland, we came to a welcome landing beyond the rugged barren mountains of North Scotland.

And the plane itself—as sturdy and buoyant and graceful as it appeared in the air—yet it was without life and entirely incapable of action unless motivated by the hand and heart and mind of a human being. Materials, alone, are not enough. Steel and plastic and rubber, that made up the plane, have no personality; copper wire and aluminum and fabrics have no memory and do not respond to incentives. Only man has been given that divine spark that transforms an organic bit of matter into a being capable of thought and love and worship.

However, if we appear in our ardent enthusiasm to overemphasize that last point, we call attention at the same time to man's definite limitations. For one thing, we humans are in fact a presumptuous lot. In the history of this world, we are now only a relatively few years advanced from the time our ancestors swung among the branches of primeval forests. Presumptuous are we also, when we boast of our scientific accomplishments, when the greatest scientific achievement of the century is one which is literally driving us underground, in unwilling retreat from our own cleverness. Presumptuous are we even in our moments of meditation when we approach the Infinite, without ourselves being able to comprehend the infinitesimal. Perhaps you have heard prayers that sound like a man telling God how to run the world instead of expressing the allegiance of one reporting for duty.

The emphasis and appreciation of people over things calls, ofttimes, for a good share of unselfishness and even humility

—dignified humility. This is found frequently among the men and women who have made noteworthy contributions in science and art and music and medicine. They have been so engrossed in the individual field in their services to humanity that they discount their own personal needs and discomforts. Many of them are interested only in worthy *ideals* and perfection of their handiwork in the service of their fellow man. They are least of all interested in material *things*. Some are not concerned whether their house furnishings go back to Louis the 14th or whether they go back to Sears & Roebuck on the 30th.

There is recognized here a seeming lack of agreement—an apparent conflict between values as they are and values as they ought to be. But, actually, there is no inconsistency. We can say with due regard for fact that we actually live in two different worlds. One of the worlds is the one we as engineers measure with transit and line and rule. The other world is the one we feel with our hearts and our imaginations. It is in this second category, strange as it may appear, that we find the origin of many of the great developments in industry and architecture and the sciences. Almost axiomatically, one cannot either devise a new process or product or construct a plant to produce it until it is clearly conceived in the imagination.

Therein lie some of the dominant differences between things and people, and the chief difference is in their relative permanency. The sensitive balance of the scientists, the transit of the engineers, and the palette knife of the artist of whatever source and of whatever value, measured solely by the yardstick of cash, are inert, without life and are valueless unless held by the hand of man. But with qualities of people—that's different. We all know that traits of character, ideals, and loyalties are transmitted from generation to generation, endlessly, across that narrow biological bridge of a single microscopic cell.

Transcending in importance all of the engineering materials and things is the welfare and development of the men who work with materials and things. And that is why we feel that to be entrusted with the supervision of other men and their work is like taking a solemn obligation—one which cannot be repudiated or laid aside. We believe, devotedly, that the greatest opportunity open to an individual is the opportunity afforded a man to grow with his responsibilities. And in this way we pay tribute to the dignity and well-being of people and to the usefulness of things. People are infinitely more important than things.

Edward Markham must have been meditating along these lines when he seized upon a basic thought and groomed it with poetic rhythm. This I leave with you in closing:

> We all are blind until we see
> That in the human plan
> Nothing is worth the making
> If it does not make the man.
>
> Why build these cities glorious
> If man unbuilded goes,
> In vain we build the world, unless
> The builder also grows.

ANNUAL REPORT OF THE BOARD OF DIRECTORS

With the activities of our Society involving such a wide range of materials and the work cutting across so many engineering fields, it is not feasible for an annual report by the directors to describe or even enumerate all phases of the work. Some indication, however, can be given of highlights of the Society's complicated operations, and these data should be recorded because the Directors and the Staff, as well as the members and committee members, have attempted earnestly to discharge our stewardship to this organization.

Appended to this report are reports from several of the administrative committees of the Society, on Research, Standards, Publications, District Activities, and Simulated Service Testing. These are worthy of study because they give a rather condensed picture of many of the Society's accomplishments.

The detailed auditors' report, also appended and supplementing the discussion on finances, covers in considerable detail this important facet of our work. It is felt that the Society is in good financial condition and that the past year was an encouraging one, indicating a continuing interest in the Society's work and persistent demand for the results of its efforts. Only by widespread dissemination and use of our standards and the information and data recorded in our technical symposiums and reports will the maximum benefits be derived from the great efforts expended by our people. It is encouraging and, in fact, one could say inspiring to note the steady increase in the use of ASTM specifications and standards.

ASTM—A Materials Society:

Many are prone, because of the great significance of our standardization activities and the widespread value coming through constant use of the standard tests and specifications, to look on ASTM as a standardization Society. In a broad sense this is the case. Yet from a broader aspect and considering the great amount of data and numerous publications coming from the widely diverse research activities carried on in the technical committees and in many other ways, one might almost say that ASTM's major forte is research in materials. Actually, of course, these two—research and standardization—frequently go hand-in-hand. Sound data are needed before specifications can be authoritatively established. Reproducible tests are essential and must be thoroughly evaluated and standardized before any specification of value can result.

From the broad viewpoint ASTM certainly has added immeasurably to our knowledge of properties and efficient use of all kinds of materials. Its research work and the technical reports and papers have had a profound effect on the whole materials field and on the development and use of new products.

Thus, while the Society's name embodies the thought of testing, down through the years the work has broadened beyond what our founders might have visualized, though indeed they were farsighted men.

We can with justification point to ASTM as a Society concerned with, and for materials.

Responsibility placed on ASTM for authoritative information and for standards on materials continues increasingly. This obligates ASTM constantly to be alert to the needs of industry and government and while it is believed through our Directors, our Administrative Committees, and especially the technical committees—the work of which is so vital in our activities—that the most important needs are soon studied and answers provided, keeping alert to the needs of industry, government, and our whole economy is one of the problems that needs constantly to be before us and this subject must be one of continuing surveillance.

Changes in Administration:

On September 16, the Board of Directors elected Robert J. Painter, formerly Assistant Secretary, and who had been serving as Treasurer since April 15, to the office of Executive Secretary. Raymond E. Hess, formerly Assistant Executive Secretary and Editor, was appointed Associate Executive Secretary and Editor-in-Chief. An announcement of the additional responsibilities of these men appeared in the October ASTM BULLETIN.

Since the Society's organization there have been two secretaries—Edgar Marburg, who served until 1918, when his assistant, C. L. Warwick, was appointed Secretary-Treasurer. The sudden death of Mr. Warwick in April, 1952, came as a great shock and in conjunction with the retirement of the Treasurer, J. K. Rittenhouse (whose death has since occurred), a heavy burden was thrown on the Headquarters' Staff. This was accentuated, too, by the preparations for the 50th Anniversay Meeting in New York. The Directors feel that they rightly can take pride in a Staff which under the circumstances carried through with so little dislocation of work and actually with a continuation and expansion of it in this period.

Death of Treasurer Emeritus; Warwick Memorial:

It is with profound regret that we record the death on March 13 of John K. Rittenhouse who was the first full-time employee of the Society and served faithfully until his retirement on April 15, 1952. His responsibilities for many years extended beyond the office of Treasurer and involved membership and meeting activities. His contributions to ASTM, his loyalty, and the friendly way in which he carried out his work will long be remembered. The Directors have adopted a suitable resolution of appreciation which has been recorded in their minutes and sent to his family.

A resolution of appreciation for the long-time Executive Secretary C. L. Warwick appeared in the 1952 Directors' Report. A special supplement to the ASTM BULLETIN is in preparation as a memorial issue, and plans are being completed for establishing a memorial room at ASTM Headquarters, containing an oil portrait, to commemorate him.

Technical Activities:

In 1952, and in some cases the early portion of 1953, many of the technical committees were completing last-minute projects so that the latest Book of ASTM Standards would be as up-to-date as possible. In one sense this seven-part 10,000-page publication stands as a monument (except that it is a very active living one) to the work of the men on the committees.

However, neither the research nor the standardization work in the committees is static, and while on the one hand some committees were completing material for the Book of Standards, a large number of new projects were initiated. Many of these are noted in the 1953 annual reports of the committees or have been covered in the news accounts given in the ASTM BULLETIN, especially the April issue, following the Spring Committee Week.

The Administrative Committee on Standards continues to handle many of the recommendations on standards which have been cleared through the technical committees. Under the sponsorship of the Administrative Committee on Research, two important documents have been issued, one a statement on some unsolved problems widely distributed to engineering schools and research groups that might be interested in some of the problems, and the second a reprint of a series of ASTM BULLETIN articles which together constitute an impressive résumé of the major research activities in the Society. No one could fail to be impressed by this document.

The committees that have been organized in the past 24 months have made splendid starts in carrying forward those segments of responsibility assigned to them. Committee E-10 on Radioactive Isotopes plans an important session and demonstration at the current Annual Meeting. Committee E-14 on Mass Spectrometry cooperated in the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy and, with other groups, arranged for a number of papers and reports in this field. Committee D-23 on Cellulose and Cellulose Derivatives is getting its program under way. The latest technical committee, concerned with soil conditioners, will function as a joint committee of ASTM and the Association of Official Agricultural Chemists. Its organization in the near future is anticipated.

A number of other fields in which it has been suggested the Society might undertake constructive standardization work are being studied. A conference on gasket materials is being arranged to review the various uses of gasketing and packing materials. ASTM, through its Technical Committee D-11 on Rubber and Rubber-Like Materials, has done important work in the field of automotive gaskets.

Technical Activities

Committee Scopes and Jurisdictions:

Committee A-9 on Ferro-Alloys:

A change in the scope of Committee A-9 on Ferro-Alloys was authorized at the request of this committee. The revised scope will now read as follows, the words in italics being new and those in brackets to be deleted:

> *Scope.*—The formulation of specifications covering *additive materials such as metals not commercially refined, ferro-alloys, and other metals and metal compounds used in melting operations in the steel and associated ferrous metal industries* [the alloys of iron with other elements commonly known as "ferro-alloys" and used commercially in the metals industry.]

Committee E-5 on Fire Tests of Materials and Construction:

In 1951 a conference of committees interested in fire tests was held to clarify ASTM work in this field and, based on this and subsequent discussion, there was adopted a set of recommendations representing the Society's policy on promulgating standards on fire tests.

In order that the scope of Committee E-5 would reflect these recommendations, the scope of this committee was clarified. Given below are both the revised scope of E-5 and the statement of policy. It is believed these will clarify this work in which the Society has made several notable contributions.

Scope.—To develop methods of tests for the determination of fire resistance and fire hazard of building materials and constructions.

To review and coordinate methods of fire tests included in standards coming within the scope of other ASTM Technical Committees.

To prepare terms and definitions for use in standard methods of fire tests of building materials and constructions.

Statement of policy

1. It is recommended that test methods for measuring combustibility characteristics and allied properties of individual materials be considered as under the jurisdiction of individual interested committees, subject however to review by Committee E-5.

2. There should be a clear understanding of the distinction between tests for various fire resistance properties of individual materials and construction assemblages. Tests of construction assemblages should be considered as solely within the scope of Committee E-5 and as the only basis sponsored by ASTM for rating the fire resistance of such assemblages.

3. The title and scope of tests for individual materials should clearly indicate the limitations on their applicability. They should be confined to developmental tests and the comparisons of the combustibility characteristics of the specific material with the view of classifying such materials with respect to their relative combustibility.

4. It should be made clear that test methods prepared for specific materials are not to be used for building code regulatory purposes when applicable methods promulgated by Committee E-5 are available.

5. It is recommended that Committee E-5 be urged to give continued and studious attention to a small scale test method for building materials and building material assemblages for use in research and development.

6. It is recommended that the title of Method E 119 (Methods of Fire Tests of Building Construction and Materials) be reviewed with a view of clarification. For example, some such wording as the following might be used:

"Standard Method of Test for Building Constructions and Materials for Fire Resistance Rating."

The small-scale panel test might be identified somewhat as follows:

"Standard Method of Test for Comparing Resistance of Building Constructions and Assemblies to Fire."

Committee C-16 on Thermal Insulating Materials:

ASTM Committee C-16 on Thermal Insulating Materials developed a revised statement of scope extending its scope of activities somewhat as indicated by the italicized words in revised statement set forth below, which has been approved by the Board:

Scope.—Formulation of test methods and specifications relating to thermal insulation materials (except those used in the insulating refractory field) *and any protective coating material associated therewith to preserve their thermal insulating efficiency.*

Patent Situations:

From time to time patent situations arise in connection with the establishment of ASTM specifications and tests. The Society has a general policy regarding these noted in the ASTM Year Book, page 598. However, each case is reviewed by the Board of Directors, consideration being given to the individual merits.

During the year patent situations have involved specifications for brazing filler metal, tests for fineness of cotton fibers by resistance to air flow, the hardness of industrial waters, and tests for measuring thickness of water-formed deposits and of corroded areas. In each case the Board has determined that in its judgment the owners of the respective patents have complied with the policies of the Society.

50-Year Anniversaries:

Three of the current technical committees of the Society were organized in 1903; thus this year will be their Fiftieth Anniversary. The standardization and research activities of these committees have been outstanding, and many notable contributions have resulted from the intensive efforts of the committee members. The committees include:

A-3 on Cast Iron
A-6 on Magnetic Properties
D-4 on Road and Paving Materials

At its October, 1952, meeting, Committee C-1 on Cement held its Fiftieth Anniversary meeting with a special technical program and ceremonies appropriate to the occasion. Its first meeting had been held in October, 1902, in Philadelphia.

Research and Standards News in ASTM Bulletin:

It is admittedly difficult to keep abreast of so many technical projects that are under way in our committees. It is suggested that one excellent way is regularly to read or scan the ASTM BULLETIN. An effort is made to present highlights of meetings of technical committees that are constantly being held, and special articles provide in a condensed form pertinent information. For example, the December, January, and February issues carried a complete review of the more than 100 research projects, giving references in each case where detailed information could be procured. A combined 24-page reprint of this material is available.

District and Related Activities

As noted in the accompanying table of ASTM meetings, most of the ASTM Districts have held meetings, many of them jointly with sections or chapters of other societies in the respective areas.

President Maxwell was greeted warmly by members and friends of the Society at the several meetings and sessions he attended. In addition to participating as the chief speaker in the Districts he visited, he also addressed the upperclassmen at a number of engineering schools, including the University of North Carolina, Rice Institute, and the University of Houston. He has also described the work of the Society at meetings of various service and civic clubs.

The accompanying report of the Administrative Committee on District Activities gives some other District news, including the organization of a new District in the Southwest. With a growing list of members and committee members there, and intensive industrial activity in many facets of which ASTM is contributing, it has been hoped that our people could participate in a District. The fine cooperation which they are giving augurs well for this latest District. The personnel of the Council will be announced in the ASTM BULLETIN.

ASTM MEETINGS, JUNE 1952–JUNE 1953, INCLUSIVE.

Meeting	Place and Date	Notes
Annual Meeting	New York, N. Y. Hotels Statler and New Yorker June 23–27, 1952	40 Technical Sessions; 450 Committee Meetings; 68 New Tentatives Approved. (Registered Attendance 2606—278 Ladies)
ASTM Committee Week	Detroit, Mich. Statler Hotel March 2–6, 1953	300 Committee Meetings (Registered Attendance 1050)
Spring Meeting	Detroit, Mich. Statler Hotel March 4, 1953	Symposium on Gloss Reception: "Fathoming the Fifties," by Kenneth McFarland.
District or Local (sponsored by Councils indicated)	Pittsburgh, Pa. University Club October 8, 1952	President's Night: "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Philadelphia, Pa. Franklin Institute October 9, 1952	Jointly with the Delaware Valley Section, Society for Experimental Stress Analysis. "Who Was the First Inventor? or Why Keep Research Records?" (discussion)
	Cambridge, Mass. (New England) Hotel Commander October 28, 1952	In association with ASTM Committees D-9 and D-20 "Plastics," by R. K. Witt and G. M. Kline
	St. Louis, Mo. Engineers' Club October 30, 1952	Joint meeting with Engineers' Club of St. Louis. "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Los Angeles. Calif. Rodger Young Auditorium November 18, 1952	Joint meeting with Southern California Section of the Society for Non-Destructive Testing, Inc. "Non-Destructive Testing," by R. C. McMaster.[b]
	Dayton, Ohio (Ohio Valley) Frigidaire Auditorium November 20, 1952	"Wood as an Engineering Material," L. J. Markwardt.
	San Francisco, Calif. St. Julien Restaurant November 25, 1952	Jointly with the Northern California Section, Society for Non-Destructive Testing, Inc. "Non-Destructive Testing," by R. C. McMaster.[b]
	New York, N. Y. Engineering Societies Bldg. December 8, 1952	"Rubber," by Gerald Reinsmith and G. C. Maasen.
	Birmingham, Ala. Tutwiler Hotel February 3, 1953	Joint meeting of Committee A-1 on Steel, ASTM; Birmingham Chapter, American Chemical Society; Birmingham Chapter, American Society for Metals; and ASTM Alabama members. "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Philadelphia, Pa. Franklin Institute February 13, 1953	President's Night: "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Berkeley, Calif. (Northern California) University of California Campus March 9, 1953	Joint meeting with California Section, American Chemical Society, and Northern California Section, American Institute of Chemical Engineers. "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Los Angeles, Calif. Rodger Young Auditorium March 12, 1953	"Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	Richland, Wash. Desert Inn Hotel March 19, 1953	Joint meeting of Columbia Basin Chapter, American Society for Metals, and Columbia Valley Section, American Institute of Chemical Engineers. "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	St. Louis, Mo. St. Louis University March 20, 1953	Jointly with American Society for Quality Control. "Some Practice in Theory and Theory in Practice," by J. H. Toulouse.
	Pittsburgh, Pa. Mellon Institute March 31, 1953	Joint meeting with Pittsburgh Section, Society of Automotive Engineers. "Titanium: Its Development and Applications," by H. T. Clark.

ASTM MEETINGS, JUNE 1952–JUNE 1953, INCLUSIVE—*Continued*

Meeting	Place and Date	Notes
	Houston, Texas Alabama Catering Service March 27, 1953	Joint meeting with American Institute of Chemical Engineers, South Texas Section. "Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a]
	New York, N. Y. Engineering Societies Bldg. April 16, 1953	"Modern Quality Control," by Simon Collier; Panel Discussion.
	Worcester, Mass. (New England) Hickory House April 16, 1953	"Chemical Developments and Engineering Materials," by President H. L. Maxwell.[a] "Applications of Abrasives," by A. L. Ball.
	Richmond, Va. (Washington, D. C.) Jefferson Hotel April 28, 1953	"Engineering in the Post Defense Economy," by A.A. Bates. "Research and Standards," by President H. L. Maxwell.
	Los Angeles, Calif. Rodger Young Auditorium June 5, 1953	"Paint and Paint Materials," by J. W. Bowen.

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[a] President Maxwell's talk, various versions of which were presented at District Meetings, was published in ASTM BULLETIN, No. 194, December, 1953, p. 47 (TP191).

[b] The address by R. C. McMaster was a condensed version of his 1952 Edgar Marburg Lecture, "Non-Destructive Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 617 (1952); also issued separately.

National Meetings

The Society's Golden Anniversary Meeting (55th Annual Meeting) was held in New York City, June 23–27, 1952. Efforts were made to make this meeting one of especial interest to foreign delegates as well as to ASTM members. The esteem in which the Society is held, and the success of this meeting in particular, is evidenced by the congratulatory expressions received from more than 150 technical and scientific societies, trade groups, and similar organizations. Sixteen foreign countries were represented. Forty technical sessions were held in addition to 450 technical committee meetings. The Apparatus Exhibit, held in connection with this meeting, was the largest ever sponsored by the Society. Including the official registration of 2606, the large numbers of visitors to the Exhibit, and those in attendance at the sessions, it is possible that upward of 5000 participated in this meeting.

The 1953 Spring Meeting, held in Detroit the week of March 2, included an outstanding Symposium on Gloss; 300 committee meetings were held during Committee Week. The mid-week dinner was highly successful due to the work of the Detroit District, which group acted as host, and to the dynamic talk by Dr. Kenneth McFarland who spoke on "Fathoming the Fifties." Detailed accounts of the 50th Anniversary Meeting and the 1953 Spring Meeting appeared in the July, 1952, and April, 1953, issues of the ASTM BULLETIN, respectively.

Centennial of Engineering:

The Society sponsored two sessions at the Centennial of Engineering in Chicago in September; N. L. Mochel delivered a paper on "Men, Materials, and the Power Industry—A Trio in Action," based in part on his 1952 Gillett Lecture. The Society coöperated in sponsoring a five-session symposium on "100 Years of Engineering Progress with Wood" by arranging for one of the sessions which was held on September 11. This was held jointly with other Societies; Committee D-7 on Wood took the lead in arranging this feature. Past-President L. J. Markwardt presided at this session, while Past-President H. H. Morgan presided at the meeting on September 10. President Maxwell represented the Society at the Centennial Luncheon and Dinner.

Abstracts of the papers have been published—Mr. Mochel's in the ASTM BULLETIN, the papers on wood in *Civil Engineering* and also as a special reprint issued by the Timber Engineering Co.

1954, 1955, and 1956 Meetings:

The 1954 Annual Meeting will be held at the Hotels Sherman and Morrison, Chicago, Ill., June 14–18. In 1955 the Annual Meeting will again return to Chalfonte-Haddon Hall, Atlantic City, N. J., during the week of June 26–July 1. The 1956 Annual Meeting is tentatively scheduled for June 17–22, also in Atlantic City.

The timing of ASTM Committee Week has been the subject of some discussion for the past several years, in view of the shortness of elapsed time between these spring group meetings and Annual Meetings and the necessity for preprinting annual reports of committees. As a result of a canvass of the various technical committees, it has been agreed that future Spring Committee Weeks should be moved up to late January or early February.

In accordance with the concensus expressed by the various committees, the 1954 Spring Committee Week will be held at the Shoreham Hotel, Washington, D. C., February 1–5. The 1955 Spring Committee Week is scheduled for the Netherlands Plaza Hotel, Cincinnati, January 31–February 4.

1956 Pacific Area Meeting:

The First Pacific Area National Meeting, held in 1949, was very successful and it was thought that another one should be held at some future date, probably in Los Angeles. Decision has been reached for the Second National West Coast Meeting to be held at the Los Angeles Statler, September 16–22, 1956. A local committee on arrangements is being organized with representatives from the Southern and Northern California Districts and other West Coast and Rocky Mountain centers. ASTM Director E. Ord Slater will be the Chairman, with W. C. Hanna, cooperating actively as Honorary Chairman; Theodore Parker Dresser, Jr., will be one of the Vice Chairmen, and Myron Niesley is Secretary. F. C. Converse is to be Chairman of the Subcommittee on Technical Program, and Claude E. Emmons is Finance Chairman. Other officers and members are to be announced.

It is expected other societies and associations may participate in some of the technical features of the meeting, and as a result of an invitation to all ASTM committees to consider West Coast meetings, a number already are planning accordingly.

Publications

The very considerable number of publications that have been issued during the past year are listed and discussed in the attached report of the Administrative Committee on Papers and Publications (see Appendix IV). Several important publication projects have reached their culmination during the past year, as for example the Fifty Year Index, the very extensive ASTM-IP Oil Measurement Tables, and the Manual on Industrial Water which, added to the considerable number of special technical publications resulting from the Fiftieth Anniversary Meeting and the appearance of the Book of Standards, created a publication schedule of quite considerable magnitude.

The special technical publications are acquiring a position of ever-increasing importance. While the Fiftieth Anniversary year was somewhat special in this respect, nevertheless, the committees are finding the sponsoring of technical symposiums very helpful in furthering the committee work and the result is a very informative and valuable series of publications.

Membership

On June 1, 1953, the membership of the Society totaled 7522, compared with a total of 7250 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1952	June 1, 1953	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	22	20	...	...	2	...	...	...	2	...	2[a]
Perpetuity and Life	11	11	...	...	...	...	...	...	...	...	...
Sustaining	255	270	1	...	...	...	15	1	1	16	15
Company, Firm, etc.	1745	1784	37	10	...	20	7	99	67	106	39
Individual, etc.	5111	5333	193	92	43	9	22	537	337	559	222
Junior	106	104	11	13	..	15	...	37	39	37	2[a]
Total	7250	7522	242	115	45	44	44	674	446	718	272
Student	398	323	18	251	...	12	...	206	281	206	75[a]

[a] Net Loss.

It will be noted that 674 new members were elected, compared with 627 for the preceding year. Although there were more losses from resignations, delinquencies, and death this year (402 compared with 378 the previous year), there is a favorable net increase of 272, compared with the net increase reported a year ago of 249. It will be seen from the table that there was a substantial increase in the number of sustaining, company, and individual memberships. The growth of the sustaining membership class is encouraging. In the past two years it has risen from 236 to the current figure of 270; and company memberships in this same period increased from 1704 to 1784. These classes particularly are important in insuring sound financial support of the Society's technical operations.

It is interesting to compare our present membership of 7522 with the figures for ten and twenty years ago; in 1943 our total membership was 4904, and in 1933 it was 3733. A true comparison would, of course, need to consider the relative percentages of sustaining, company, and individual memberships. Nevertheless, the steady growth in the total membership is encouraging.

Sustaining Members:

There have been sixteen new sustaining members, and there was one resignation. The new sustaining members are:

American Chemical Paint Co.
American Phenolic Corp.
Catalin Corporation of America
The Cleveland Twist Drill Co.

Colgate-Palmolive-Peet Co.
Erie Railroad Co.
B. F. Goodrich Chemical Co.
Hanson-Van Winkle-Munning Co.
Lehigh Portland Cement Co.
The Lummus Co.
The Maytag Co.
The Midvale Co.
National Research Council of Canada
Niagara Mohawk Power Corp.
Rohm & Haas Co.
Sandia Corp.

50-Year Members:

There are nine individuals and companies as noted below that have been connected with the Society continuously for 50 years, having joined in 1903:

Frederick W. Bateman
Almon H. Fuller
Herbert F. Moore
C. P. Van Gundy
L. W. Walter
The Carpenter Steel Co.
Arthur D. Little, Inc.
National Lead Co.
Pennsylvania State Highway Dept.

These members will be recognized at the annual meeting.

40-Year Members:

There is a total of 37 members who this year have completed 40 years of membership, which brings the total number of certificates issued to the "Forty-Year Club" to 251. The members who will receive the 40-Year Certificates at this annual meeting are as follows:

American Car & Foundry Co.
Louis Anderson
The Atlantic Refining Co.
John M. Bierer
A. Burton Cohen
Combustion Engineering, Inc.
Compressed Gas Mfrs. Assn., Inc.
Cork University College Library
John J. Crowe
Henry Disston & Sons, Inc.
Electro Metallurgical Div., Union Carbide & Carbon Corp.
Dudley K. French
Giant Portland Cement Co.
Dean Harvey
Hydro-Electric Power Commission of Ontario
Burgis D. Jennings
Arthur M. Johnsen
Leeds & Northrup Co.
Missouri State Mining Experiment Station, Ceramics Div.
Northern Pacific Railway Co.
The Ohio Brass Co.
Oliver Iron & Steel Corp.
Otis Elevator Co.
The Pennsylvania Railroad Co.
Julian A. Pollak
University of Queensland
Ralph E. Roscoe
Rutgers University, School of Ceramics
Seattle, City Engineer's Dept.
Henry T. Shelley
The A. P. Smith Mfg. Co.
Tennessee Coal & Iron Div., U. S. Steel Co.
City of Toronto, Dept. of Public Works
Valentine & Co., Inc.
C. Benson Wigton
L. H. Winkler
Harry Wolf

Deaths:

The Society has lost the following 45 members through death (figures in parentheses are dates of membership):

Buchholtz, H. (1940)
Buck, Harold J. (1940)
Carder, C. H. (1937)
Carlson, T. A. (1937)
Carver, Fred S. (1929)
Cooper, Howard (1946)
Devereux, W. C. (1931)
Eavenson, Howard N. (1921)
Fairbairn, John MacFarlane (1938)
Fox, Edwin H. (1950)
Greiner, Leonard E. (1948)
Grossmann, Marcus A. (1925)
Hallett, R. L. (1920)
Haven, George B. (1917)
Johnston, Robert S. (1919)
Lee, Alan Porter (1936)
MacGregor, John R. (1912)
Matte, Joseph, Jr. (1943)
McKesson, Claude L. (1930)
McManus, J. D. (1943)
*Miller, J. Strother (1903)
Oldacre, William H. (1932)
Pleister, Henry W. (1926)

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* Honorary Member.

Poorman, Alfred P. (1908)
Quinn, Don L. (1937)
Reilly, Edmund A. (1925)
Rosenthal, Helman (1927)
Sanders, Vic (1945)
Schaeffer, Emil (1951)
Schreiber, F. D. (1952)
Shepard, Hugh Martin (1943)
Smith, Paul H. (1947)
Sparrow, Stanwood W. (1930)
Stinson, T. K. (1940)
Sunderland, Charles C. (1925)
Thrasher, Grafton M. (1939)
Tinsley, John F. (1912)
Tomkins, Selah S. (1920)
van der Graaf, J. (1951)
*von Shrenk, Hermann (1903)
Waterhouse, George B. (1922)
Wilbur, David T. (1949)
Wilson, Hewitt (1933)
Wiskocil, Clement T. (1916)
Withrow, James R. (1929)

In addition to the above, the following representatives of company members of the Society passed away:

George E. Bean, The Eastern Malleable Iron Co.
O. F. Blakey, University of Western Australia
Harold A. Dambly, Philadelphia Electric Co.
G. B. Gould, Fuel Engineering Co. of New York
Otto E. Hager, Chesapeake & Ohio Railway Co.
E. C. Page, Page Testing Laboratory
Morris Schrero, Carnegie Library of Pittsburgh
W. W. Scott, Jr., The Laclede Steel Co.
H. J. Wernert, Wernert, Taylor, Sanzenbacher, & Morris

A number of these men have been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The notable work and meritorious service of two of the men, J. Strother Miller and Hermann von Schrenk, had been recognized through election to Honorary Membership in the Society. Dr. von Schrenk was a Past-President of the Society, a prime mover in the organization of Committee D-7 on Wood, and its chairman for 44 years; he was also one of the organizers of the St. Louis District Council and its first Chairman (1939–1946). Mr. Miller, a leader in the asphalt technology field, made many important and varied contributions in technical committees, his activities being concentrated chiefly in Committees D-4 on Road and Paving Materials and D-8 on Bituminous Waterproofing and Roofing Materials, but also extending into Committees C-1 on Cement and D-1 on Paint, Varnish, Lacquer, and Related Products. Both Messrs. Miller and von Schrenk would have been in the ASTM "Fifty-Year" group this year.

T. A. Carlson, of the Forest Products Laboratory, worked actively in Committee D-6 on Paper and Paper Products, and for many years in Committee D-10 on Shipping Containers where he had been Chairman for the past six years. M. A. Grossman, an outstanding authority in the field of metallurgy, served on several ASTM committees in the ferrous field and on Committee E-9 on Fatigue, and was particularly active in Committee E-4 on Metallography where he directed work involving grain size that brought widespread recognition to the Society. In the death of R. L. Hallett (after several years' retirement), the Society loses one who had done much constructive work in Committee D-1 on Paint, having been Secretary for ten years. He also had served on Committee B-2 on Non-Ferrous Metals and Alloys and as a Director of the Society, 1935–1937. George B. Haven, a leader in the field of textile technology, concentrated his ASTM work in Committee D-13 on Textile Materials, serving as Chairman from 1917 to 1920, and later being elected to honorary membership in this group. Robert S. Johnston, for many years Director of Research, John A. Roebling's Sons Co., served on several ASTM committees in the field of steel, corrosion, and screen wire cloth.

John R. MacGregor, a 40-year member of the Society, was a leader in work on protective coatings, and served for many years on Committee D-1 on Paint. Claude L. McKesson had been active in the work of Committees D-4 on Road and Paving Materials and D-18 on Soils for Engineering Purposes. William H. Oldacre and S. W. Sparrow participated in the work of Committee D-2 on Petroleum Products and Lubricants. Don L. Quinn participated actively in the work of the Committees on Paper and Paper Products, and Shipping Containers. Vic Sanders was a member of the Pittsburgh District Council and also of Committee C-16 on Thermal Insulating Materials. S. S. Tompkins served on several ASTM committees concerned with fuels—D-3 on Gaseous Fuels and D-5 on Coal and Coke—and also on D-16 on Industrial Aromatic Hydrocarbons and D-19 on Industrial Water. George B. Waterhouse, distinguished for his research work in iron and steels, had served on various ASTM committees, principally A-1 on Steel, A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys, and E-8 on Nomenclature and Definitions. Hewitt Wilson was concerned especially with the activities of Committees C-8 on Refractories and C-21 on Ceramic Whiteware. James R. Withrow, longtime Professor of Chemical Engineering, Ohio State University, directed the work of ASTM Committee C-7 on Lime as Chairman for a number of years and was a member of Committee C-12 on Mortars.

Concerning those men who represented their companies in the Society, exercising the responsibilities of membership, George E. Bean served on Committee A-7 on Malleable Iron Castings and several subcommittees. In the sudden death of Harold A. Dambly, of the Philadelphia Electric Co., the Society lost one who, although active in ASTM a relatively short time, had done very constructive work in the Philadelphia District. A member of Committee D-5 on Coal and Coke and a number of its subgroups for many years, G. B. Gould was Chairman of the Subcommittee on Significance of Tests. A former member of the St. Louis District Council, W. W. Scott, Jr., served for over 25 years on Committee A-1 on Steel.

Former Active Members:

Harry J. Love, for many years Secretary and Managing Director of the National Slag Assn., was active in the Society for over 30 years and was a loyal and active member of Committees C-9 on Concrete and Concrete Aggregates and D-4 on Road and Paving Materials. He was an honorary member of Committee C-9 and was active in various associations in the Washington, D. C., area.

Frank M. Waring, formerly Engineer of Tests, Pennsylvania Railroad Co., and for many years retired, was a former Chairman and Secretary of Committee A-1 on Steel and served on several other committees, including D-11 on Rubber where he had been Secretary. He was a former Director of the Society.

More detailed notes on the work and contributions of many of these men have appeared in respective issues of the BULLETIN. The Society records its deep appreciation for their contributions and the spirit which motivated them.

Honors and Awards

In line with recently established practice, the Board has continued recognition for outstanding service through the selection of members to receive the

Award of Merit. Various awards and medals are also given to recognize outstanding papers and to foster research in certain special fields.

Award of Merit:

The Award of Merit Committee for 1953, comprising H. M. Hancock, chairman, T. S. Fuller, A. T. Goldbeck, H. W. Stuart, and W. A. Zinzow, reviewed numerous suggestions and citations from various technical committees and nominated ten members who by unanimous action of the Board were voted the 1953 Awards of Merit. Each one has contributed in an important way to the work of the Society. Award of Merit certificates will be presented during this Annual Meeting to the following:

Everett G. Ham
James T. MacKenzie
Dalton G. Miller
Rudolph E. Peterson
Carlton H. Rose
Walter A. Selvig
Erle I. Shobert II
Earl R. Stivers
Jerome Strauss
Roderick B. Young

Recognition of Technical Papers:

The following awards will be made at this Annual Meeting in recognition of outstanding technical papers:

Charles B. Dudley Medal—presented for a paper of outstanding merit constituting an original contribution on research in engineering materials. Medals this year will be presented to E. A. Davis and M. J. Manjoine of Westinghouse Research Laboratories, East Pittsburgh, Pa., for their paper on "Effect of Notch Geometry on Rupture Strength at Elevated Temperatures."

Richard L. Templin Award—presented for a paper describing new testing methods and apparatus, the purpose of the award being to stimulate research in the development of testing methods and apparatus. This year the award will be made to W. N. Findley and P. G. Jones, University of Illinois, W. I. Mitchell, South Dakota School of Mines, and R. L. Sutherland, University of Iowa, for their paper on "Fatigue Machines for Low Temperatures and for Miniature Specimens."

Sam Tour Award—presented for a paper describing new and useful testing procedures, apparatus, or new and useful evaluation of previously developed corrosion testing methods, the purpose of the award being to encourage research on the improvement and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field. This year the award will be made to J. R. McDowell of Westinghouse Research Laboratories, East Pittsburgh, Pa., for his paper on "Fretting Corrosion Tendencies of Several Combinations of Materials."

Sanford E. Thompson Award—presented by Committee C-9 on Concrete and Concrete Aggregates for a paper of outstanding merit in the field of concrete and concrete aggregates. The award this year will be presented to Mrs. Katharine Mather, Chief, Petrography Section, Concrete Research Division, Waterways Experiment Station, Corps of Engineers, Jackson, Miss., for her paper on "Applications of Light Microscopy in Concrete Research."

The Harold Dewitt Smith Memorial Medal was presented to Herbert J. Ball, Head of the Department of Engineering, Lowell Textile Institute, Lowell, Mass., at the March 19 meeting of Committee D-13 on Textile Materials. This medal is awarded for outstanding achievement in the field of textile fiber science and utilization. A past-president of the Society, Professor Ball was

Chairman of Committee D-13 for many years.

Frank E. Richart Award:

The Board has authorized a Frank E. Richart Award, established by a gift from Mrs. Richart, to honor and perpetuate the memory of her late husband, who was a long-time member of the Society, a member and officer of various technical committees, a Vice-President, and Honorary Member of the Society. The award is to encourage work related to the activities of Committee C-9 on Concrete and Concrete Aggregates (where Professor Richart was most active) and to recognize outstanding contributions made in this field. The recipient of the award will be named periodically, but not more often than every third year, and will receive a certificate and cash.

New Board Committee on Lectures and Awards:

By action of the Board of Directors in September, 1952, a special committee consisting of G. R. Gohn, chairman, L. C. Beard, and M. A. Swayze was appointed to coordinate the planning for all Society lecturers and awards and to set up rules of procedure for securing lecturers with the thought of providing adequate time for the necessary contacts. On the recommendation of the special committee, the Board has appointed from its members a Committee on Lectures and Awards, whose continuing responsibility is the establishment and administration of rules and regulations with respect to the selection of memorial lecturers and recipients of the various awards. This committee will review for the Board the reports of the various lecture and award committees, will make recommendations to the Board for appointments to the committees selecting the lecturers and award winners, and generally coordinate activities of this nature.

Finances

Report for the Fiscal Year of 1952:

The annual statement of the finances of the Society is presented in the form of the report of the auditors for the fiscal year January 1 to December 31, 1952, which appears in Appendix I. The report gives the balance sheet of assets and liabilities, including general funds, building fund, and other special and designated funds; a statement of receipts and disbursements classified into "operating" (budgeted) and "non-operating"; details of the building fund and of special funds; and investments of Society funds.

Operating Receipts:

Total operating receipts were $648,352.34, the largest in the Society's history. Of this amount receipts from dues and entrance fees were $226,283.99 or 34.9 per cent; receipts from sales of publications were $338,447.83 or 52.2 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, registration fees, and exhibit, total $83,620.52 or 12.9 per cent.

Income from sales of publications was just under the 1951 record high, which is significant. Considering that the Society's financial operations should be studied really on a three-year basis because of the triennial publication of our Book of Standards which provides our largest sales income, it is encouraging to note the large income in the third year of this period. (Heaviest income for the 1949 Book of Standards was in 1950.) In 1952 operations the income from 1950 and 1951 Standards Supplements, from special compilations of standards, and so-called separates, and especially from miscellaneous publications (symposiums, charts, etc.) bulked large in the sales income.

Operating Disbursements:

With a steadily increasing number of standards and continuing demand for them, more technical symposiums than ever before, and with considerably higher publication and other costs, our total disbursements were much higher than in 1951, and for any other year, totalling $620,030.26. The budget recognized that this situation would probably develop. It is worthy of note that our actual disbursements were about $9000 under the estimate. Publications costs, exclusive of overhead, labor, etc., totalled $259,822.59, or 41.9 per cent of the total. Salaries accounted for 34.1 per cent of total expenditures, which is virtually the same ratio as in recent years.

Considering the nature and extent of the Society's 50th Anniversary Meeting in New York, with several special events, it is of interest that our costs were well under the modest budget of $9500. The New York Committee on Arrangements did excellent work in raising a special fund of some $20,000 to defray the cost of special entertainment features, but including also laboratory and plant trip programs, various printing and promotional costs, and the photographic exhibit.

The Society's contribution to the Retirement Plan for ASTM employees was $16,013, about 2.6 per cent of total disbursements and about 7.5 per cent of the salary roll. The Social Security Tax, included in this total, was $2314. Further discussion of the Retirement Plan is given on a later page.

Summary of 1952 Operations:

Comparing operating receipts with disbursements, there was a favorable balance of $28,322.08. This was encouraging since a normal third year after publication of the Book of Standards might result in a deficit and budget operations must be based on the possibility of such a situation.

Balance Sheet.—The appended auditors' report includes a balance sheet of assets and liabilities as of December 19, 1952. Assets in the General Funds totalled $568,557.72 compared to $524,490.65 a year ago. Total assets including the Building Fund and Special and Designated Funds are $1,004,216.76 compared to $947,676.30 a year ago. This increase in assets reflects substantial increase in cash and investments in the General Funds and in Special Funds.

It should be pointed out that in the Balance Sheet no account has been taken of the assets of the Society in the form of publications in stock. This is consistent with many years' practice in this respect and, of course, results in a more conservative statement. An inventory of the principal technical publications as of May 1, 1953, is summarized below:

Proceedings (All back copies not incl. 1952)	2 133
1952 Book of ASTM Standards	
Part 1	6 287
Part 2	5 285
Part 3	8 036
Part 4	4 053
Part 5	4 570
Part 6	4 022
Part 7	4 314
ASTM Methods of Chemical Analysis	2 030
Special Compilations of Standards	18 652
Selected Standards for Students	19 830
Symposiums and Special Reprints	120 285
Reprints of Standards (Approx.)	200 000
Radiographic Standards for Steel Castings	14

The surplus in General Funds is $265,824.26 which compares with $259,063.13 a year ago.

ASTM Building Fund.—The auditors' report contains a statement of receipts and disbursements and the Balance Sheet for the ASTM Building Fund. The Building Fund received from General Funds the sum of $5907.34 to cover payment of interest and amortization of the loan. The Fund also received $5700 for depreciation account which was paid in monthly installments and invested in shares of Commonwealth Investment Co., a mutual fund. Other receipts of $2499.16 constituted contributions from members and interest and dividends on investments.

The Balance Sheet is set up to show as assets the complete purchase price of the Headquarters' Building and construction costs from the beginning (depreciated in accordance with the agreed-upon schedule) and all contributions to the Fund as a liability. The reserve for depreciation, $31,450, added to the Building Fund from General Funds for the past five years, appears in the Balance Sheet as a deduction from the construction costs. The Society's headquarters, including the cost of the Cherry Street lot purchased in 1952 at a cost of $29,179, represents an asset on the depreciated basis of $184,811.50.

To simplify the Building Fund operations, the two notes outstanding on the Headquarters' Building and the Cherry Street lot were combined in 1952 and are now being amortized at a rate to clear them in five years.

It should be noted, too, that the Finance Committee, cognizant of the studies on Headquarters' Building expansion, has transferred to the General Funds all of the Building Fund assets represented by the Commonwealth Fund, and has deposited an equivalent amount in Savings Funds in the interest of liquidity.

Investments.—Investments of Society funds (General, Building, Research, Committee, Medal and Lecture Funds) as of December 19, 1952, are given in the appended auditors' report, both book and market values being shown. As of that date the book value of all investments was $392,450.47, compared with $361,915.06 a year ago; the market value of all investments on December 19, 1952, was $421,020.50.

The following changes in investments during the calendar year 1952 are listed as a matter of record:

GENERAL FUNDS

Sold		*Proceeds*
	Bonds	
$20 000	Federal Intermediate Bank Deb. (matured)	$20 000.00
	Preferred Stocks	
20 sh.	Philco Corp. 3¾%	3 748.52
	Common Stocks	
100 sh.	Baldwin Lima Locomotive	1 043.51
100 sh.	Baldwin Securities	388.84
50 sh.	Dow Chemical Rights	22.71
100 sh.	Lone Star Cement Co.	2 472.60
100 sh.	International Harvester Co.	3 275.62
	Total Preferred Common and Bonds	$30 951.80

Bought		*Cost*
	Bonds	
$15 000	U. S. Treasury 2¼% 1955–52	$15 004.69
15 000	U. S. Treasury 2% 1954–52	14 943.75
15 000	U. S. Treasury 2% 1954–52	14 962.50
	Common Stocks	
60 sh.	Jones & Laughlin Steel Co.	5 386.73
	Mutual Funds	
26 sh.	Fundamental Investors Inc. (Capital Gain)	503.62
9 sh.	Broad Street Investing Corp. (Capital Gain)	191.97
49 sh.	Eaton & Howard Balanced Funds (Capital Gain)	1 497.44
9 sh.	Commonwealth Investing Co. (Capital Gain)	61.74
	Total Bonds Common and Mutual Funds	$52 552.44

BUILDING FUND

Bought		*Cost*
	Mutual Funds	
138.927 sh.	Commonwealth Investing Co. (dividend reinvested)	$952.79
761.005 sh.	Commonwealth Investing Co. (1952 depreciation)	5 700.00
	Total Mutual Funds	$6 652.79

RESEARCH FUND

Sold		*Proceeds*
	Preferred Stocks	
20 sh.	Philco Corp. 3¾%	$1 667.70
29 sh.	Consumers Power $4.50	2 970.18
12 sh.	Consolidated Edison $5	1 279.12
	Total Preferred Stocks	$5 917.00

Bought	*Cost*
Mutual Funds	
340 sh. Eaton & Howard Balanced Funds	$11 310.19
9 sh. Broad Street Investing Corp. (dividend reinvested)	191.97
Total Mutual Funds	$11 502.16

TEMPLIN FUND

Bought	*Cost*
Mutual Funds	
1 sh. Eaton & Howard Balanced Funds (dividend)	$30.56

TOUR FUND

Bought	*Cost*
Mutual Funds	
1 sh. Eaton & Howard Balanced Funds (dividend)	$30.56

1953 Budget:

In the budget for 1953, current income has been placed at $716,500, made up of dues and entrance fees of $229,000; publications sales, $430,500; miscellaneous items, $57,000.

Estimated disbursements are $701,700, made up of publication costs of $285,700, salaries (including Staff expansion and increases in salaries appropriate to meet current conditions), $248,300; and general office expenses, meetings and committees, headquarters, retirement, and miscellaneous, $167,700.

Comparing estimated income and expenditures, a favorable balance of $15,000 is expected. Actually it is hoped this will be considerably greater because this is the first year of our triennial budgeting period, and we normally look to a favorable balance to aid in carrying through the leaner second and third years. The Finance Committee believes the budget reflects a conservative attitude, with income estimated somewhat on the low side and disbursements somewhat on the high side.

To complete reference to 1953 finances thus far this year, it is noted that during the first four months the following changes in investments have taken place:

GENERAL FUNDS

Bought	*Cost*
*15 sh. Commonwealth Investment @ $6.99	$104.85
*45 sh. Eaton & Howard Balanced Fund	1 440.90
*5 sh. Broad Street Investing Corp.	112.05
*21 sh. Fundamental Investors, Inc.	425.46
8 sh. Gulf Oil Corp. (dividend)	

BUILDING FUND

Bought	*Cost*
66.225 sh. Commonwealth (Multiple Purchase Acct.)	500.00
*77.823 sh. Commonwealth	543.98

RESEARCH FUND

Bought	*Cost*
*6 sh. Broad St.	134.46
*7 sh. Eaton & Howard	224.14

TEMPLIN AWARD FUND

Bought	*Cost*
*1 sh. Eaton & Howard	32.02

TOUR AWARD FUND

Bought	*Cost*
*1 sh. Eaton & Howard	32.02

COMMITTEE FUNDS

Bought	*Cost*
$20 000 U. S. Treasury 2S 12/15/54–53 (X-ray Diffraction)	19 925.00

* Capital Gains Dividend Shares.

The total book value of Society investments as of March 23, 1953, in all funds was $415,925. The market value was $444,682, divided approximately into 39.4 per cent, bonds, 10.8 per cent, preferred stocks, and 49.8 per cent, common stocks. The Society's cash balance in General Funds as of the same date was $376,103.

Retirement Plan and Social Security

The ASTM Retirement Plan instituted May 3, 1938, has been amended a few times since then, the last amendment becoming effective this year. The purpose of this plan is to provide a retirement benefit or pension for participating employees who remain in the employ of the Society until retirement date.

In addition to this plan the employees are covered by the Social Security Law, benefits from which will be in addition to the Retirement Plan. ASTM employees contribute a percentage of their salary both to the Retirement Plan and to Social Security.

The pension income formula provides that the retiring employee receive a yearly pension equal to 1 per cent of his average yearly base salary multiplied by years of service. The average salary would be that for his five highest base yearly salaries, but excluding any portion of salary in excess of $6000. This latter figure was changed in 1953 from $5000. The Finance Committee felt that this change would provide a more realistic program and ameliorate some of the effects of inflation. Each employee affected will increase his contribution. (By this change in the Plan, an employee with 30 years of service on retirement would have his income increased by 0.30 times $1000, or $25 per month.)

At the end of 1952 there were 27 participants in the Plan, and two additional employees will enter it in May, 1953. A number of additional retirement policies are being taken out on the May 3 anniversary date covering employees affected by the amendment noted above or by changes in salaries.

The Directors have authorized a Group Life Insurance Plan for the Staff employees with a policy of $1000 each. Upon retirement, the employee will have the option of continuing this policy at his own expense, without any health examination. At the same time there will be instituted a hospitalization plan on a group subscription basis. The cost to the Society of these benefits will total about $1400 annually.

Including Social Security, it is expected the Society's total retirement and benefit cost for 1953 will aggregate about $20,000, which represents about 8.1 per cent of the salary budget.

Administrative Matters

As mentioned earlier in the report, Mr. R. J. Painter has been appointed as Executive Secretary to succeed Mr. C. L. Warwick. R. E. Hess was appointed Associate Executive Secretary and Editor-in-Chief, continuing his position as Technical Secretary. Mr. Painter continues to serve as Treasurer.

There have been a number of other changes in the Society's Staff.

John S. Pettibone, a member of the Staff for several years and concerned with varied technical work, including especially the various projects on corrosion, and with recent added responsibility for District Activities, has been advanced to Assistant Technical Secretary.

Samuel F. Etris has joined the Technical Staff. A graduate in chemistry, with his master's degree in ceramics, he will be concerned particularly with some of our committee work in the "C" and "D" Groups.

In April, William F. Black was appointed Technical Editor. Mr. Black returned to the Staff after a period of service in industry. He will be concerned largely with development and editing of technical papers, symposiums, etc., including editorial work on the ASTM BULLETIN and miscellaneous assignments where his editing facilities will be most helpful. Sheldon E. Fitterer, formerly of the Standards Department, who has had experience in advertising and marketing, was transferred to work in the field of advertising and developmental activities.

We are fortunate in being able to maintain a loyal Staff. Every department has had increasing loads during 1952 and these have been intensified during the first months of 1953, the 1952 Book of Standards and the unusually large number of technical publications and compilations creating greatly increased publication, accounting, and sales-order activities.

Society Headquarters:

In late 1951, the Society acquired a lot at the rear of the present Headquarters' Building which has about twice the area of the original purchase. The steadily growing work of the Society necessitating an increase in technical staff operations, the handling of more publications and orders, and the necessity for additional space for records, business machine facilities, and related equipment, is taxing the present building facilities. A special committee of the Board has been examining into the best means of making the necessary additional facilities available. Conferences have been held with the Moore Institute of Art and the Academy of Natural Sciences, our immediate neighbors in the block. An architect is engaged in studies which may include preliminary plans for expansion.

Details of the building fund are given in another section of this report. The Headquarters' Building and the Cherry Street lot are carried on the balance sheet as an asset of $184,811.50 after depreciation. The Finance Committee is accelerating the amortization of the properties at a rate which would pay off the notes outstanding on them in about five years.

Cooperative Activities

The Society has many cooperative activities with other technical and scientific groups. Many of these have been under way for years and carried out as regular procedure. A few of these follow: the work of Technical Committee A on Automotive Rubber (a Joint SAE-ASTM project); the Joint Committee on Filler Metal (an American Welding Society-ASTM activity); Joint Committee on Effect of Temperature (ASME-ASTM project); Joint Committee on Paper Testing Methods (TAPPI-ASTM); Paint, Varnish, and Lacquer (Federation of Paint and Varnish Production Clubs-ASTM). There are a number of other joint committees listed in the ASTM Year Book, and there is given also a list of Society representatives in other activities, including sectional committees organized under the auspices of the American Standards Association, many of which are sponsored by the Society. Each year the Society representatives are asked to submit reports on matters of moment that are of concern to ASTM. The time devoted to this work is another indication of the splendid support ASTM receives from its members and representatives. The work of our representatives and their reports are very effective in coordinating technical work and avoiding to a considerable extent duplication of effort. With such a tremendous load being carried by the engineering fraternity today, it seems important that our efforts be concentrated and that there be as little duplication of effort as possible. This has been a consistent policy of the Society since its inception.

Another example of a cooperative arrangement involves the new committee on soil conditioners where the Association of Official Agricultural Chemists will sponsor a joint committee which will be organized promptly to develop necessary data and subsequently to develop methods of evaluation for these relatively new materials which are coming into widespread use. ASTM work in this field was authorized at the September meeting of the Board.

The Society is vitally interested in the work of the Standards Division of the Federal Services of Supply, which operates under the General Services Administration of the Federal Government, and also in the work of the Defense Supply Management Agency which was created to coordinate the testing and specification requirements of the Armed Services. Two conferences were held in Washington with responsible individuals in these groups—the first in the office of Clifton E. Mack, Commissioner of the Federal Services of Supply, with W. S. MacLeod, Chief, Standard Division, and other Government men present. The second conference was a joint one with representatives of the Standards Division, Federal Services of Supply, and the Defense Supply Management Agency. Because of legal problems which may be involved in coordinated effort, representatives of legal staffs were in attendance. The ASTM representatives included a special committee of the Board of Directors on Government contacts headed by N. L. Mochel, with Messrs. Schatzel and Anderton present at the second conference. Representatives of the American Standards Association attended the joint conference also. (It is of interest that if the plan of the President and his advisers becomes effective, the Munitions Board, Research and Development Board, and the DSMA would be discontinued, although in the case of the latter the work would continue under an Assistant Secretary of Defense, and with this setup the work on cataloging and standards coordination could be strengthened and expedited.)

There is no question of the desirability of coordinating such specifications and tests as are issued by the Society and other recognized standard bodies (many of which may have received approval by ASA) with the requirements of the military and civilian branches of the Government. In fact, many ASTM standards are now embodied in Federal requirements, for example, in the testing of petroleum. The Society has indicated its earnest desire to cooperate fully in any rationalization approach. Some of the ASTM technical committees are now aiding and advising appropriate Government agencies. It is the strong desire of industry, and unquestionably beneficial to Government as well as industry, to have effective coordination, and the current administration has indicated its support of this ideal.

In any discussion of this involved problem, reference should be made to the outstanding cooperation that has existed for many years on the part of many of the civilian and military branches of the Government in supporting the work of the Society along technical as well as administrative lines.

Ordnance Advisory Committee:

There has been set up for some time an ASTM Ordnance Advisory Committee to cooperate in some of the problems of the Ordnance Department of the Army. Panels of this group hold periodic meetings, these covering such subjects as adhesives, plastics, and rubber.

During the year, there was a clarification of the legal status of this committee, and since the members have indicated their willingness to continue service and since ordnance officials desire it, the directors are recommending that the committee continue. Essentially this is a special committee and does not fall into the category of an industry advisory committee.

Office of Defense Mobilization:

The 1952 Report of the Directors detailed an arrangement that had been set up with the Office of Defense Mobilization by which J. R. Townsend, supplemented by an engineer at ASTM Headquarters, gave advice and assistance to the ODM on matters concerning conservation of materials. Several conferences that were held on such matters as nickel and cobalt were detailed in the May and September 1952 issues of the ASTM BULLETIN. The contract with ODM was terminated on September 1, 1952.

American Standards Association:

The Society has continued its active support of the work of the American Standards Association in many technical and administrative lines. The accompanying report of the Administrative Committee on Standards (Appendix II) gives some detail of our relations with the ASA.

A large number of ASTM standards have received approval as American standard through the various methods that can be used in submitting recommendations. Once a specification or test has been approved by ASA, there is the matter of keeping it up to date. Both a new submittal and the submission of revisions involve considerable time and effort. This problem is receiving the study of the appropriate ASA and ASTM officials. One proposal that would expedite the work is that the ASTM Administrative Committee on Standards be designated to function as a reviewing committee of ASA to consider any ASTM standards submitted for ASA approval. This matter is still being studied. Despite the widespread recognition given to the ASTM standards as such, approval by ASA does give some added prestige and recognition and further publicity results.

K. G. Mackenzie was appointed an ASTM representative in the Standards Council, and he serves with J. R. Townsend and J. H. Foote, our two other representatives. Other ASTM appointments on ASA committees include the following: L. B. Jones reappointed to ASA Mechanical Standards Committee; R. C. Thumser reappointed to ASA Chemical Industry Correlating Committee; G. H. Harnden reappointed to Miscellaneous Projects Correlating Committee; J. H. Foote appointed to succeed F. M. Farmer on ASA Electrical Standards Committee.

Other appointments on Sectional Committees have been listed in the ASTM BULLETIN or under the section on Appointments in this report.

International Activities:

A summary of the contacts of the Society with international standardization activities, especially those in the International Organization for Standardization (ISO), appears in the appended report of the Administrative Committee on Standards. References are made to work in the following fields: textiles, petroleum products, solid mineral fuels, rubber, viscosity, plastics, shellac, iron and steel, chemistry, laboratory glassware, terms on chemical and physical test results, mica, copper and copper alloys, paper, and timber. Several ASTM committees handle the American contacts with ISO committees, and America holds the secretariat for some of these ISO groups.

The Society will be represented at the International Soils Conference to be held in Zürich in August. The ASTM representatives will be W. G. Holtz, U. S. Bureau of Reclamation, and Gregory P. Tchebotarioff, Princeton University.

The Society during the year welcomed many visitors from abroad, and several of the teams sponsored by the Mutual Security Agency have visited ASTM Headquarters.

Appointments:

The following appointments made during the year relate to our cooperative activities:

J. H. Foote, Commonwealth Services, Inc., to succeed F. M. Farmer, on the ASA Electrical Standards Committee;

L. B. Jones, Consulting Engineer, reappointed to ASA Mechanical Standards Committee;

R. C. Thumser, Monsanto Chemical Co., reappointed to ASA Chemical Industry Correlating Committee;

G. H. Harnden, General Electric Co., reappointed to Miscellaneous Projects Correlating Committee;

H. S. Meissner, U. S. Bureau of Reclamation, on ASA Sectional Committee A-1 on Hydraulic Cements, succeeding H. S. Mattimore, Miller-Warden Associates;

R. E. Hess, Associate Executive Secretary, ASTM, on ASA Sectional Committee A-39 on Window Cleaning;

L. D. Betz, W. H. & L. D. Betz, succeeding F. N. Speller, Metallurgical Consultant, on the Joint Research Committee on Boiler Feedwater Studies;

R. J. Painter, Executive Secretary, ASTM, on Executive Committee of Conference of Executives of ASA Member Bodies (CEOM);

R. J. Painter, to the Council of the American Association for the Advancement of Science;

H. J. Ball, Lowell Textile Institute, reappointed to ASA Consumer Goods Committee;

Walter M. Scott, U. S. Department of Agriculture, as an additional ASTM representative on the Joint ASTM-AATCC Committee on Textile Test Methods;

W. J. Krefeld, Columbia University, for J. W. Whittemore, Virginia Polytechnic Institute, on ASA Construction Standards Board;

J. S. Nelson, Monsanto Chemical Co., for H. K. Nason, Monsanto, on ASA Sectional Committee Z26 on Safety Glass;

C. A. Baker, U. S. Testing Co., on ASA Sectional Committee Z66 on Hazards to Children;

K. G. Coutlee, Bell Telephone Laboratories, on ASA Sectional Committee C55 on Capacitors, succeeding E. O. Hausmann, Continental-Diamond Fibre Co.;

R. E. Hess, Associate Executive Secretary, ASTM, to succeed C. L. Warwick, deceased, on ASA Sectional Committee A-21 on Cast Iron Pipe, serving as Treasurer;

K. G. Mackenzie, Texaco Development Corp., to succeed C. H. Rose, National Lead Co., on ASA Standards Council;

P. M. Brister, Babcock & Wilcox Co., as a member of the Joint Committee on Effect of Temperature on the Properties of Metals.

Respectfully submitted on behalf of the Board of Directors,

H. L. MAXWELL,
President.

R. J. PAINTER,
Executive Secretary.

June, 1953.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1 TO DECEMBER 19, 1952

Philadelphia, January 9, 1953

MR. R. J. PAINTER, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the period ended December 19, 1952, the date the society closed its books for the year 1952. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts.

Cash on deposit in checking and savings accounts was verified by direct mail confirmation with the various depositories and reconciled with the cash records. Accounts receivable were not verified by correspondence with debtors. The investments owned by the Society, as detailed on pages 9 to 12, were examined by us. Such securities are held in safe deposit box of the Society at the Girard Trust Corn Exchange Bank. Income from investments for the period under review was verified by us and properly recorded on the books of the Society.

We have prepared and submit herewith balance sheet as of December 19, 1952, statement of cash receipts and disbursements for the period ending that date, and other supporting schedules which, in our opinion, subject to the foregoing comment, present fairly the financial position of the Society on December 19, 1952, and the results of its operations for the period ended that date.

Respectfully submitted,

JOHN HEINS & CO.

BALANCE SHEET AS OF DECEMBER 19, 1952

(Including Special and Designated Funds)

ASSETS

General Funds:			
Cash	$283 537.56		
Less Check No. 1203 as of December 17, 1952 (drawn against cost of 1952 *Proceedings* and 1952 Book of Standards)	83 000.00		
	200 537.56		
Investments (Market Value $306 052.75)—Cost	282 493.36		
Accounts Receivable	26 550.85		
Notes Receivable (Building Fund)	36 000.00		
Advance to Forest Products Laboratory	500.00		
Total Current Assets General Funds		$546 081.77	
Furniture and Fixtures (depreciated book value)		22 475.95	
Total Assets General Funds			$568 557.72
Building Fund:			
Cash		1 719.29	
Investments (Market Value $32 054.98)—Cost		33 132.31	
Land (Cost)		30 000.00	
Building and construction costs	156 390.87		
Less Reserve for depreciation	31 450.00	124 940.87	
Land and improvements, 1915–19 Cherry Street, Philadelphia, Pa.		29 870.63	
Total Assets Building Fund			$219 663.10
Other Special and Designated Funds:			
Cash: ASTM Research Fund	5 685.36		
Dudley Medal and Marburg Lecture Fund	1 419.91		
Richard L. Templin Award Fund	83.06		
Sam Tour Award Fund	109.00		
Committee Funds	131 873.81	139 171.14	
Investments:			
ASTM Research Fund (Market Value $61 345.53)—Cost	53 637.06		
Dudley Medal and Marburg Lecture Fund (Market Value $5 327.50)—Cost	6 625.00		
Richard L. Templin Award Fund (Market Value) $1 089.02)—Cost	1 025.18		
Sam Tour Award Fund (Market Value $1 633.53)—Cost	1 537.56		
Committee Funds (Market Value $13 517.19)—Cost	14 000.00	76 824.80	
Total Assets, Special and Designated Funds			$215 995.94
Total Assets			$1 004 216.76

Note.—All market values of securities are as of December 19, 1952.

LIABILITIES

General Funds:			
Advance Collections	$10 793.46		
Accounts Payable:			
Committee C-1	142.63		
Joint Committee on X-ray Diffraction	75 019.36		
Institute of Petroleum	551.52		
Total Current Liabilities, General Funds		$86 506.97	
Life Membership Fund	3 812.50		
Publication Fund	16 841.80		
Book of Standards Reserve	101 900.18		
Executive Retirement Reserve	15 992.60		
Retirement Fund Reserve	24 042.77		
Reserve for Depreciation of Investments	20 000.00		
Reserve for Additional Cost of Replacement of Headquarters Building	25 000.00		
Special Annual Meeting Entertainment Account	8 586.64		
Warwick Memorial	50.00	216 226.49	
		302 733.46	
Surplus		265 824.26	
Total Liabilities and Surplus, General Funds			$568 557.72
Building Fund:			
Contributions from Members		171 627.80	
Contributions from ASTM General Funds		10 969.57	
Accumulated Income, Profit on Investments		1 065.73	
Notes Payable to ASTM General Fund		36 000.00	
Total Building Fund			$219 663.10
Other Special and Designated Funds:			
ASTM Research Fund:			
Principal	53 671.45		
Income	5 650.97	59 322.42	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	1 419.91	8 044.91	
Richard L. Templin Award Fund:			
Principal	1 037.12		
Income	71.12	1 108.24	
Sam Tour Award Fund:			
Principal	1 537.56		
Income	109.00	1 646.56	
Committee Funds, Unexpended Balances		145 873.81	
Total Special and Designated Funds			$215 995.94
Total Liabilities and Funds			$1 004 216.76

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1948–1952 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1948	$50 691.65	$191 497.99	$46 748.11	$17 261.45	$7 777.78	$40 000.00	$31 260.92	$227 160.50
1949	56 379.22	192 325.57	36 382.87	18 078.39	12 349.77	40 000.00	31 580.92	219 235.36
1950	147 957.36	222 662.96	50 692.18	17 959.00	59 919.59	61 900.18	65 973.89	251 477.84
1951	164 497.75	263 995.13	77 239.16	18 758.61	72 990.86	101 900.18	90 536.48	259 063.13
1952	200 537.56	282 493.36	63 050.85	22 475.95	86 506.97	101 900.18	114 326.31	265 824.26

COMMITTEE FUNDS

Committee A-1 On Steel	$1 577.24	
Committee A-5 On Corrosion of Iron and Steel	1 109.85	
Committee B-3 On Corrosion of Non-Ferrous Metals and Alloys	2 534.83	
Committee B-6 On Die-Cast Metals and Alloys	2 697.56	
Committee B-8 On Electrodeposited Metallic Coatings	11.25	
Committee C-1 On Cement (General Funds)	1 655.61	
Committee C-1 Cement Reference Laboratory	633.79	
Committee C-9 Sanford E. Thompson Medal Fund	1 016.32	
Committee C-9 On Concrete and Concrete Aggregates	151.17	
Committee C-15 On Manufactured Masonry Units	404.33	
Committee C-16 On Effect of Moisture in the Performance of Thermal Insulation	6 845.00	
Committee C-18 On Natural Building Stones and Slate	398.99	
Committee D-1 On Paint, Varnish, Lacquer, and Related Products	528.45	
Committee D-2 On Petroleum Products and Lubricants	783.80	
Committee D-2 ASTM-IBM	903.79	
Committee D-2 D.C.C. Reference Fuel Account	21 026.72	
Committee D-2 D.C.C. National Exchange Group	2 356.35	
Committee D-2 D.C.C. Equipment Development Project	348.70	
Committee D-4 On Road and Paving Materials	6.95	
Committee D-5 On Coal and Coke	1 561.06	
Committee D-7 Pole Research Fund	1 839.02	
Committee D-9 On Electrical Insulating Materials	2 085.60	
Committee D-10 On Shipping Containers	19.49	
Committee D-11 On Rubber and Rubberlike Materials	81.75	
Committee D-12 On Soaps and Other Detergents	489.75	
Committee D-13 On Textile Materials	1 121.19	
Committee D-15 On Engine Antifreezes	43.00	
Committee E-3 On Chemical Analysis of Metals	159.54	
Committee E-9 On Fatigue	204.31	
ASA Sectional Committee on Specifications for Cast Iron Pipe	1 391.40	
Detroit District	3 175.68	
Philadelphia District	425.58	
Philadelphia District (Annual Meeting Account)	711.31	
Pittsburgh District	525.85	
Western New York—Ontario District	225.72	
Advisory Committee on Corrosion	70 272.31	
Administrative Committee on Ultimate Consumer Goods	208.30	
Joint Committee on X-ray Diffraction	11 175.53	
Joint Committee on X-ray Diffraction Research Associate	5 166.72	
		$145 873.81
Accounted for as follows:		
Cash in bank and on hand	131 873.81	
Investments (at cost)	14 000.00	
		$145 873.81

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1952:			
Principal—Investments (at cost)	$6 625.00		
Income—Cash	1 569.91	8 194.91	
Receipts:			
Interest on investments		330.00	8 524.91
Disbursements:			
Honorarium		400.00	
Dudley Medal		38.50	
Miscellaneous (Travel exp., etc)		41.50	480.00
			8 044.91
Accounted for as follows:			
Principal—Investments (at cost)		6 625.00	
Income—Cash in bank		1 419.91	$8 044.91

RICHARD L. TEMPLIN AWARD FUND

	Total	Invested	Uninvested Cash
Principal Account:			
Balance, January 1, 1952	1 006.56	994.62	11.94
Receipts:			
Transfer from income	30.56		30.56
Disbursements:			
Purchase of securities		+30.56	−30.56
	1 037.12	1 025.18	11.94
Income Account:			
Balance, January 1, 1952		55.37	
Receipts:			
Interest and dividends on investments		72.56	127.93
Disbursements:			
Transfer to principal account		30.56	
Templin Awards		25.00	
Engrossing certificates		1.25	56.81
Balance, December 19, 1952			$71.12

SAM TOUR AWARD FUND

	Total	Invested	Uninvested Cash
Principal Account:			
Balance, January 1, 1952	1 507.00	1 507.00	
Receipts:			
Transfer from income account	30.56		30.56
Disbursements:			
Purchase of securities		+30.56	−30.56
	1 537.56	1 537.56	0.00
Income Account:			
Balance, January 1, 1952		45.75	
Receipts:			
Interest and dividends on investments		93.81	139.56
Disbursements:			
Transfer to principal account			30.56
Balance, December 19, 1952			$109.00

ASTM RESEARCH FUND

	Total	*Invested*	*Uninvested Cash*
Principal Account:			
Balance, January 1, 1952	$48 504.34	$48 313.79	$190.55
Receipts:			
Sale of investments	−261.89	−6 178.89	5 917.00
From Building Funds	330.00		330.00
Transfer from Income Account	5 099.00		5 099.00
Disbursements:			
Purchase of securities		+11 502.16	−11 502.16
	53 671.45	53 637.06	34.39
Income Account:			
Balance, January 1, 1952		9 034.63	
Receipts:			
Interest and dividends on investments		2 223.76	11 258.39
Disbursements:			
Transfers to principal account		5 099.00	
Welding Research Council		250.00	
Printing, etc.		258.42	5 607.42
Balance, December 19, 1952			$5 650.97

ASTM-ODM FUND

Balance, January 1, 1952	0.00	
Receipts:		
Advance from general funds	2 500.00	
Cash from United States Government	6 105.11	8 605.11
Disbursements:		
Salaries	5 103.00	
Expenses	1 002.11	
Return of advance to general funds	2 500.00	8 605.11
Balance, December 19, 1952		$0.00

RECEIPTS AND DISBURSEMENTS

For the Period January 1, 1952 to December 19, 1952

Cash balance, January 1, 1952			$164 497.75
Receipts			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$216 367.60		
Past Dues	1 147.32		
Advance Dues	4 329.07		
Income, Life Membership Fund	175.00		
Entrance Fees	4 265.00		
Total Dues		$226 283.99	
Sales of Publications:			
Book of Standards (Members, additional parts)	54 919.35		
1949 Book of Standards	39 300.53		
1952 Book of Standards	3 271.45		
1950 Supplements to Book of Standards	17 341.05		
1951 Supplements to Book of Standards	46 148.98		
Methods of Chemical Analysis of Metals	5 010.42		
Compilation of Standards	60 561.48		
Separate Standards	23 501.75		
Selected Standards for Students	4 883.95		
Proceedings	6 795.88		
50 Year Index and Papers	4 280.85		
Spring Meeting Papers	1 441.50		
Bulletin Subscriptions (non-members)	3 093.14		
Special and Miscellaneous Publications	67 897.50		
Total sales of publications		338 447.83	
Miscellaneous:			
Advertising—Bulletin	28 734.42		
Advertising—Index	1 872.50		
Interest and Dividends	19 263.72		
Registration and Other Fees—Meetings	14 277.30		
Annual Meeting Exhibit	16 242.75		
Staff Services	2 723.97		
Miscellaneous	505.86		
Total Miscellaneous Items		83 620.52	
Total Operating Receipts (Budgeted)		648 352.34	
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	30 967.31		
Excess Remittance	2 562.83		
Committee C-1 for Technical Assistant	2 780.00		
Retirement Fund Reserve	15 980.59		
Executive Retirement Reserve	504.60		
Joint AIME-ASTM Publication Fund	75.25		
Sale of X-ray Diffraction Cards	42 906.25		
Institute of Petroleum Publications	473.74		
From Building Fund on Account of Loan	4 778.61		
Annual Meeting Luncheons	1 280.00		
Return of Advance to ODM Project	2 500.00		
Special Annual Meeting Entertainment Account (Atlantic City)	8 586.64		
Annual Meeting Dinner and Buffet Supper	3 366.75		
Warwick Memorial	50.00		
Accrued Interest	263.84		
Total Nonoperating Receipts		117 076.41	
Total Receipts			765 428.75
Total Receipts for 1952 and Cash Balance, January 1, 1952			$929 926.50

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year book	$12 098.63		
1949 Book of Standards	1 836.69		
1952 Book of Standards	65 238.37		
1951 Supplements to Book of Standards	6 053.15		
Emergency Alternate Standards	1 362.24		
Compilation of Standards	26 033.87		
Separate Standards	11 404.35		
1951 Proceedings	874.32		
1952 Proceedings	35 153.52[a]		
Preprints	9 390.85		
Spring Meeting Papers	815.15		
ASTM BULLETIN	31 842.47		
Circular to Members	2 644.28		
50 Year Index to Papers	4 435.81		
Index to Standards	8 101.20		
Special and Miscellaneous Publications	42 537.69		
Total disbursements—Publications		259 822.59	
Salaries	211 816.89		
General Office Expenses	60 475.47		
Expenses—Technical and District Committees	11 827.30		
Expenses—Meetings	19 507.54		
Headquarters Occupancy Expense (includes $5 700 depreciation on building)	24 001.50		
American Standards Association	1 750.00		
Traveling Expense, Administrative and Special Committees	6 487.93		
Furniture and Fixtures	6 928.19		
Dues, Contributions, Miscellaneous	400.07		
Legal Services	1 000.00		
Employees' Retirement Fund	13 698.62		
Federal Old Age and Survivors	2 314.16	360 207.67	
Total Operating Disbursements (Budgeted)		620 030.26	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	2 562.83		
Investments	52 822.79		
Technical Assistant Committee C-1	2 668.68		
Transfer to Joint Committee on X-ray Diffraction	20 000.00		
Cost and Refunds on X-ray Diffraction Cards	15 250.86		
Transfers to New York Committee on Arrangements	3 557.54		
Annual Meeting Luncheon	1 933.98		
Advance to ASTM-ODM Project	2 500.00		
Executive Retirement Fund	1 332.00		
Gillette Memorial	430.00		
Gift to J. K. Rittenhouse	500.00		
Miscellaneous	5 800.00		
Total Nonoperating Disbursements		109 358.68	
Total Disbursements			729 388.94
Cash Balance, December 19, 1952			$200 537.56

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[a] These accounts include $83,000 representing a check drawn against cost of the 1952 Proceedings and 1952 Book of Standards, but not actually paid on December 19, 1952.

ASTM BUILDING FUND

DETAILS OF ASTM BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1952 TO DECEMBER 19, 1952

Cash balance, January 1, 1952			$1 572.28
Receipts			
Contributions from Members		878.87	
Interest and Dividends on Investments		1 290.29	
Rent from 1915 Cherry Street		330.00	
From ASTM General Fund:			
On Account of Depreciation on Building, Elevator and Air Conditioning Installation		5 700.00	
On Account of Interest and Amortization of Note		5 907.34	14 106.50
			15 678.78
Disbursements			
Purchase of 899.932 shares Commonwealth Investment Co.		6 652.79	
Contributions to ASTM Research Fund[b]		330.00	
Taxes on Cherry Street Property		377.34	
Improvements to Cherry Street Property:			
Fence	387.00		
Signs	40.00		
Quarry Street entrance	265.02	692.02	
Interest on Loan from ASTM General Fund		1 128.73	
Reduction of Loan from ASTM General Fund		4 778.61	13 959.49
Cash Balance, December 19, 1952			$1 719.29

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[b] This represents rent from 1915 Cherry Street.

ASTM BUILDING FUND

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF DECEMBER 19, 1952

Assets

Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa.				$1 719.29
Investments: (Market value $32 054.98)				
$8 500 U. S. Treasury Bonds 2½s 1967–72			8 632.83	
5 000 U. S. Savings Bonds Series G 2½s			5 000.00	
2 746 405 Shares Commonwealth Investment Co.			19 499.48	33 132.31
Property 1916–18 Race Street, Philadelphia, Pa:				
Purchase Price	53 625.00			
Commission to Agent	2 681.25			
Settlement Fee, net	197.10			
	56 503.35			
Allocated to:				
Land			30 000.00	
Building		26 503.35		
Construction Costs:				
Architect's Fees	11 544.70			
Rent Paid to Facilitate Possession	110.00			
Zoning Fees and Permits	17.50			
General Contractor	86 933.72			
Air Conditioning	20 749.00			
Elevator	7 548.04			
Air Conditioning Unit	650.75			
Exhaust System for Basement	216.25			
Real Estate Agent's Fee	500.00			
Soundproofing	1 953.00			
Miscellaneous	817.48			
	131 040.44			
Less Proceeds from Sales and Salvage of Equipment and Rentals	1 152.92	129 887.52		
		156 390.87		
Reserve for Depreciation (provided from General Funds)		31 450.00	124 940.87	
			154 940.87	
Property 1915–19 Cherry Street, Philadelphia, Pa:				
Purchase Price	27 500.00			
Commission to Agent	1 375.00			
Settlement Fee, net	303.61	29 178.61		
Improvements:				
Fence	387.00			
Signs	40.00			
Quarry Street entrance	265.02	692.02	29 870.63	184 811.50
Total Assets				$219 663.10

Liabilities and Funds

Contributions from Members			$171 627.80	
Contributions from ASTM General Funds			10 969.57	
Accumulated Income:				
Interest and Dividends on Investments		4 357.76		
Profit on Disposal of Securities, net		76.27		
		4 434.03		
Less Interest on Loan from General Fund	2 990.96			
Real estate taxes on Cherry Street	377.34	3 368.30	1 065.73	
Notes payable to ASTM General Funds			36 000.00	
Total Liabilities				$219 663.10

INVESTMENTS

AS OF DECEMBER 19, 1952

GENERAL FUNDS

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
$4 000	Erie R.R. Co. Gen. Conv. Income "A" 4½s 1/1/2015	$2 832.00	$3 230.00
37 600	U. S. Savings Bonds Series G 2½s	37 600.00	36 058.80
5 000	Walworth Co. 3¼s Conv. Deb. 5/1/76	5 496.53	3 837.50
15 000	U. S. Treasure 2¼s 6/15/55-52	15 004.69	14 995.31
15 000	U. S. Treasury 2s 12/15/54-52	14 943.75	14 906.25
15 000	U. S. Treasury 2s 6/15/54-52	14 962.50	14 934.38
		$90 839.47	$87 962.24
Shares	*Preferred Stocks*		
25	Allis-Chalmers Mfg. Co. 3¼s cumulative convertible	$2 220.60	$3 012.50
67	American Airlines, Inc. 3½s cumulative convertible	5 911.55	5 360.00
50	Chicago, Rock Island & Pacific R.R. Co. 5s "A" cumulative convertible	4 434.60	4 681.25
50	Food Machinery and Chemical Co. 3¼s cumulative convertible	4 750.10	5 106.25
60	Jones and Laughlin Steel Corp. 5% cumulative "A"	5 386.73	5 010.00
		$22 703.58	$23 170.00
	Common Stocks		
100	Allis-Chalmers Mfg. Co., no par value	$4 170.75	$6 050.00
100	American Rad. & Std. Sanitary Corp.	1 564.32	1 412.50
25	American Telephone & Telegraph Co.	3 798.72	3 925.00
100	American Tobacco Co.	7 370.97	6 550.00
60	American Viscose Corp.	2 323.58	3 495.00
100	American Cyanamid Co.	3 669.14	5 375.00
50	Aluminum Co. of America	3 679.74	4 675.00
115	Carpenter Steel Co.	6 104.75	5 189.38
100	Chase National Bank	3 412.50	4 775.00
15	Delaware Power & Light Co.	235.00	375.00
156 33/40	Shs Dow Chemical Co.	3 944.32	6 757.50
100	General Telephone Corp.	3 312.82	3 512.50
200	Gulf Oil Corp.	6 273.27	9 975.00
200	International Harvester Co.	6 406.67	6 400.00
200	Lone Star Cement Co.	4 941.44	6 075.00
45	Montgomery, Ward & Co.	2 646.41	2 756.25
100	Ohio Oil Co.	2 623.35	5 550.00
200	Philadelphia Electric Co.	4 352.72	6 450.00
125	Public Service Electric & Gas Co.	4 558.50	3 359.38
50	Republic Steel Corp.	2 152.93	2 250.00
100	Socony-Vacuum Oil Co.	2 510.28	3 625.00
30	United Gas Improvement Co.	567.50	1 005.00
		$80 619.68	$99 537.51
	Mutual Funds		
266	Broad Street Investing Corp.	$5 262.99	$6 006.28
534	Commonwealth Investment Co.	3 552.99	3 716.64
2 046	Eaton & Howard Balanced Fund	61 670.77	65 533.38
990	Fundamental Investors, Inc.	17 843.88	20 126.70
		$88 330.63	$95 383.00

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[a] Market values were taken from current financial publications as of close of market December 19, 1952.

ASTM BUILDING FUND

Par Value	Bonds	Cost or Book Value	Market Value
$8 500	U. S. Treasury 2½s 6/15/72–67	$8 632.83	$8 160.00
5 000	U. S. Savings Bonds, series G 2½s	5 000.00	4 780.00
		$13 632.83	$12 940.00

Shares	Mutual Funds		
746 405	Commonwealth Investment Co.	$19 499.48	$19 114.98

ASTM RESEARCH FUND

Par Value	Bonds		
$1 500	B. & O. R.R. Ref. & Gen. 5s, Series M 3/1/96	$1 511.25	$1 220.63
9 900	U. S. Defense Savings Bonds, Series G 2½s	9 900.00	9 529.20
2 000	U. S. Treasury 2½s 3/15/70–65	2 000.00	1 928.13
		$13 411.25	$12 677.96

Shares	Preferred Stocks		
30	Allis-Chalmers Mfg. Co. 3¼s cumulative convertible	$3 007.22	$3 615.00
33	American Airlines, Inc. 3½s cumulative convertible	3 366.00	2 640.00
		$6 373.22	$6 255.00

Shares	Common Stocks		
100	Allis-Chalmers Mfg. Co.—no par value	$4 421.59	$6 050.00
196	General Motors Corp.—Gift[a]	6 504.75	13 328.00
100	Jones & Laughlin Steel Corp.	2 636.06	2 250.00
55	Montgomery, Ward & Co.	3 151.63	3 368.75
		$16 714.03	$24 996.75

Shares	Mutual Funds		
289	Broad Street Investing Corp.	$5 828.37	6 525.62
340	Eaton and Howard Balanced Fund	11 310.19	10 890.20
		$17 138.56	$17 415.82

DUDLEY MEDAL AND MARBURG LECTURE FUND

Par Value	Bonds	Cost or Book Value	Market Value
$500	B. & O. R.R. Ref. & Gen. Mtg., 6s Series J, 1995	$550.00	$460.00
6 000	B. & O. R.R. Ref. & Gen. Mtg., 5s Series G, 1995	6 075.00	4 867.50
		$ 6 625.00	$5 327.50

RICHARD L. TEMPLIN AWARD FUND

Shares			
34	Eaton and Howard Balanced Fund	$1 025.18	$1 089.02

SAM TOUR AWARD FUND

Shares			
51	Eaton and Howard Balanced Fund	$1 537.56	$1 633.53

COMMITTEE FUNDS

Par Value			
$1 000	U. S. Treasury Bonds 2⅞s 1960–55	$1 000.00	$1 027.50
2 000	U. S. Treasury Bonds 2½s 1969–64	2 000.00	1 937.50
3 000	U. S. Treasury Bonds 2½s 1970–65	3 000.00	2 892.19
3 000	U. S. Treasury Bonds 2½s 1972–67	3 000.00	2 880.00
5 000	U. S. Treasury Bonds Series G 2½s	5 000.00	4 780.00
		$14 000.00	$13 517.19

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[a] The income from these shares is, by agreement, paid to an annuitant.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards has, in accordance with its regulations, carried out over the year various activities. Its principal function, however, has been the passing upon tentatives and revisions which have been submitted to it. Hereinafter is included a complete record of such recommendations as have been approved.[1] These actions include 30 new tentatives accepted during the year as well as revisions of 69 tentatives, tentative revisions of 10 standards, 8 standards reverted to tentative, 9 standards and tentatives withdrawn, and the withdrawal of emergency alternate provisions in 2 standards. A large number of these were acted upon at meetings of the committee held on September 5, 1952, and December 12, 1952, and the remainder were handled by correspondence.

The committee has continued its efforts toward improvement of quality and form of standards. At the instigation of the Standards Committee, editorial committees have been organized by a number of the technical committees. Also at the instance of the committee, the Board of Directors has arranged with Committee E-1 for preparation of a manual on the form of ASTM methods for guidance of the technical committees. It is planned that when this manual is completed it will be put in the hands of subcommittee and group leaders engaged in the writing of methods of testing as well as in the hands of technical committee chairmen and officers. It is believed this will aid the committees in the treatment of certain important features such as editorial form, accuracy, reproducibility, etc.

The committee had submitted to it during the year the question of having a recognized definition of a classification since several classifications presently appear in the Book of Standards although this category is not specifically referred to in the Regulations Governing Technical Committees. The following definition was accordingly prepared by the committee for incorporation in the Regulations:

> "A classification as applied to materials or products may be defined as a systematic arrangement or division into groups based on similar characteristics such as origin, composition, properties or use."

EXPANSION OF STANDARDIZATION ACTIVITIES

Gasket Materials.—Consideration is being given to the desirability of expanding the work which has been done in the Society on gaskets, through the formation of a new committee to cover sundry gasketing and packing materials. A conference on this subject is being arranged at which it is proposed to review the various uses of gasketing and packing materials and the extent of coverage that should be included, also the type of materials.

Soil Conditioners.—A new committee on soil conditioners has been authorized by the Board of Directors with Prof. W. P. Martin of Ohio State University as temporary chairman. It is planned to set this up as a joint committee of the ASTM and the Association of Official Agricultural Chemists in view of the latter's interest in identification tests for soil conditioners. The interest of ASTM is in the evaluation or performance tests.

Other Pending Proposals.—At various times inquiries have been received and proposals have been submitted with respect to the Society undertaking work in the field of ore sampling, surface chemistry, abrasives, water-repellent solutions for masonry surfaces, ion exchange, osmotic diaphragms, autoclave pressure field, and heavy chemicals.

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[1] This list is not reproduced here; the recommendations have all been announced currently throughout the year in the ASTM BULLETIN.—Ed.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Assn. are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship so far as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations but with ASTM as sponsor or co-sponsor for the sectional committee.

Standards Submitted under the Existing Standards Procedure:

The following standards were submitted to the American Standards Assn. for approval as American Standard under the existing standards procedure:

Standard Spec. for Building Brick (Solid Masonry Units Made from Clay or Shale) (C 62 – 50), and

Standard Spec. for Facing Brick (Solid Masonry Units Made from Clay or Shale) (C 216 – 50).

Revised Standards Submitted under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Assn. during the year revisions of one specification relating to malleable iron castings, 9 relating to copper and brass materials, 3 standards relating to gypsum, 9 relating to manufactured masonry units, 4 relating to rubber and rubber products, 2 relating to paint pigments, 1 relating to rosin, and 2 relating to soap.

Standards Submitted under Sectional Committee Procedure:

During the year, reports were received from four of the sectional committees for which the Society is sponsor or co-sponsor as follows:

Sectional Committee A88 on Magnesium Oxychloride Cement Flooring.— This committee has recommended the approval as American Standard of Specifications for Non-Spark Conductive Oxychloride Composition Flooring and Its Installation.

Sectional Committee C7 on Bare Electrical Conductors.—This committee has recommended the approval of revisions of 29 American Standards.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval of revisions of 13 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 4 ASTM Methods, and the approval of revisions of 22 American Standards.

All of these reports have been referred to the American Standards Assn. Action is still pending so far as the recommendations of Sectional Committee A88 and Sectional Committee L14 are concerned; the others have been approved.

INTERNATIONAL RELATIONS

The Society continues to receive from other countries standards with the request for comment. ASTM committees review these standards and frequently suggestions result. Apart from such informal cooperation, however, quite a number of proposals have been received through the American Standards Assn. concerning projects that have been proposed under the International Organization for Standardization (ISO).

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on Textiles but America holds the secretariat for several subcommittees. American participation in the work of ISO/TC 38 is being handled by a special committee organized in the American Standards Assn. under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists, with representation from the various groups interested. This special committee has been given the designation of "L23."

Meetings were held in New York in June, 1952, of the subcommittees on shrinkage of fabrics in washing, and on yarn testing, as well as the working group on cloth strength testing using the grab technique. The subcommittee on colorfastness met in New York in November, 1952.

Petroleum Products.—America holds the secretariat for ISO/TC 28 on Petroleum Products, the work of the secretariat being handled by Sectional Committee Z11, of which ASTM is sponsor.

A meeting of ISO/TC 28 was held in New York City in June, 1952, and the committee adopted as its scope of work methods of sampling, methods of test, nomenclature, terminology, and specifications for petroleum and petroleum products.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. ASTM Committee D-11 is handling the American contacts with this ISO committee. A meeting of ISO/TC 45 is being held in Paris, France, June 15–20, 1953, at which it is expected America will have a number of delegates.

Viscosity.—America holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 is being looked to to handle the work of the secretariat.

Plastics.—America holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 is being looked to to handle the work of this secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

A meeting of ISO/TC 61 is being held in Stockholm, Sweden, August 10–14, 1953, at which it is expected America will have a number of delegates.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat. America has submitted a number of comments with respect to standards dealing with shellac that are now under consideration.

A meeting of ISO/TC 50 was held in New York City in June, 1952.

Solid Mineral Fuels.—ASTM Committee D-5 is handling American contacts with ISO/TC 27 on Solid Mineral Fuels. A meeting of the Special Working Group on Volatile Matter of ISO/TC 27 was held in April, 1953, at which America was represented by Mr. R. F. Abernethy of the U. S. Bureau of Mines.

A meeting of ISO/TC 27 is scheduled

for London, England, on October 5–9, 1953.

Iron and Steel.—A meeting of ISO/TC 17 on Iron and Steel, was held in New York in June, 1952. Consideration was given to draft proposals on Brinell Hardness Test, Rockwell Hardness Test, Vickers Hardness Test, Bend Test, Charpy Impact Test, and Izod Impact Test. Members of ASTM Committee A-1 attended this meeting (as observers) together with members of Committee E-1 subcommittees on Indentation Hardness and Impact Testing.

Chemistry.—The following ASTM committees have indicated an interest in the work of ISO/TC 47 on Chemistry:

D-1 on Paint, Varnish, Lacquer, and Related Products
D-3 on Gaseous Fuels
D-12 on Soaps and Other Detergents

Laboratory Glassware.—Committee E-1 has indicated that it favors American participation in the work of ISO/TC 48 on the basis of an observer.

General Definitions Relating to Chemical and Physical Test Results.—ASTM Committee E-11 favors American participation in the work of ISO/TC 69. A meeting of this Committee was held in Geneva, Switzerland, on November 26–30, 1951, which was attended by W. Edwards Deming, member of ASTM Committee E-11 on Quality Control.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 has been set up to handle the American contacts with this ISO Committee 56 for which India holds the secretariat. There is now before the committee three standards dealing with mica which evolved from the meeting of ISO/TC 56 held in New York City in June, 1952.

Copper and Copper Alloys.—America has been invited to take the secretariat for ISO/TC 26 on Copper and Copper Alloys. This subject is of interest to ASTM Committee B-5, and consideration is now being given as to how to handle the American participation in the work.

Paper.—On the recommendation of ASTM Committee D-6 on Paper and Paper Products America participates in the work of ISO/TC 6 on the basis of an observer. Copies of all proposals are received and referred to Committee D-6 for its information.

Timber.—ASTM Committee D-7 on Wood has indicated that it favors American participation in the work of ISO/TC 55 on Timber.

Respectfully submitted on behalf of the committee,

L. B. JONES,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research held one meeting during the year, at Society Headquarters on December 29, 1952.

While the present research fund is not sufficiently large to underwrite any extended research program, nevertheless the attention of the various technical committees is being called to the availability of the Administrative Committee on Research for help, in connection with research problems, in the following ways:

(*a*) In assisting in the raising of funds to carry out a project,

(*b*) In providing funds for a specific piece of equipment, or

(*c*) For the purpose of standardizing a piece of equipment between several cooperating laboratories.

A trial mailing of a pamphlet entitled "Some Unsolved Problems" was made to 87 deans of engineering, chemistry and graduate schools, etc., at 45 colleges and universities. On the basis of replies received from more than 50 per cent of the schools approached, a new mailing recently has been made to all accredited schools in this country and Canada; more than 1900 letters have been sent out. The purpose of this mailing is to bring before educational institutions the interest of ASTM in research as well as to provide subjects for possible research work. This latest printing of "Some Unsolved Problems" contains more than 50 problems contributed by 13 of the Society's committees. Distribution to research institutions and commercial laboratories is currently being planned.

To ascertain the rate of progress for the solution of these unsolved problems, the various committees will be circularized possibly a year or two hence, requesting which of the problems now classified as unsolved have had a solution which is acceptable to the committee directly concerned with the problem. It is proposed also that some four or five years from now, copies of "Some Unsolved Problems" will be sent to those included on the original mailing list asking the recipients to indicate which problems have been either partially or wholly completed, following up this information with a request for sources of any pertinent papers or related information.

A "Review of ASTM Research" appeared in the December, 1952, and January and February, 1953 BULLETINS. This review supplements reviews of 1947, 1943, and earlier years. Since publication in the BULLETIN, this review has been made available as a separate reprint.

It is encouraging to report that the Joint Committee on Effect of Temperature on the Properties of Metals has passed the three-quarter mark in its solicitation for a fund of $85,000 to be used for elevated temperature research on metals. Committees B-4 and E-3 are cooperating on the development of methods for chemical analysis of cathode nickel. Development of these methods is complicated by the necessity for a relatively large sample for analysis as opposed to the cost. In the case of finished cathodes which are very light in weight, several hundred cathodes are needed for a sample. However, progress has been made and it is hoped that five methods may be approved by the Joint Committee before the end of this year. Committee C-16 on Thermal Insulating Materials has raised half of the estimated necessary fund of $15,000 for research on the determination of effect of moisture on thermal conductivity, and a contract has been entered into with The Pennsylvania State College for a part of this work. The wood pole testing program sponsored by Committee D-7 on Wood has reached the solicitation stage and is being brought to the attention of interested groups. The committee is desirous of obtaining a sum in the order of $150,000 to be used in the testing of full-sized poles on the correlation of tests of full sized poles with the usual laboratory-size specimens.

The Society has continued its relations with the Division of Engineering and Industrial Research of the National Research Council, and the activities of the Highway Research Board and the Building Research Advisory Board of this Division have been of considerable interest to the Society. Suggested research projects have been discussed with the Public Housing Administration and the Reunion des Laboratoires d'essais et de Recherches sur les Materiaux de les Constructions.

Respectfully submitted on behalf of the committee,

W. C. VOSS,
Chairman.

J. S. PETTIBONE,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

At no time has the Committee on Papers and Publications been faced with a publication problem of the magnitude of that which it had to handle during the past year. Not only is this a Book of Standards year involving the publication of a book much larger than heretofore, but a number of other special publications of considerable magnitude were published as well. The number of technical papers has been abnormally large this year as a result of the Fiftieth Anniversary Meeting held in New York City last June. The many sessions held as a part of this meeting resulted in 15 special technical publications, and a number of other papers have been published in the ASTM BULLETIN and in the 1952 *Proceedings*. In all, the Papers Committee reviewed and accepted for publication 227 technical papers.

One volume of special interest is the Fifty Year Index (covering the years 1898 to 1950) which was gotten out somewhat as a commemorative volume for the Fiftieth Anniversary. The style heretofore used in the decennial index was considerably changed so that the combined index is of an easily usable size. In preparing this volume, the committee had the advice of several librarians—Mr. Iver Igelsrud, Battelle Memorial Inst., Mrs. Marie S. Goff, E. I. du Pont de Nemours & Co., and Miss Leah E. Smith, Bell Telephone Laboratories, Inc.

The work that had been under way for several years came to fruition in the publication of the extensive volume of tables entitled ASTM-IP Petroleum Measurement Tables. This had involved a very large amount of calculation and represented an outstanding piece of cooperative work with the Institute of Petroleum Technologists.

During this same year, a new edition was issued of the ASTM Manual of Engine Test Methods for Rating Fuels; a cumulative publication of the Bibliography on Electrical Contacts; and a cumulative publication of the Metal Cleaning Bibliography. Also worthy of special mention is the Manual on Industrial Water, a publication on which Committee D-19 has been at work for some time and which represents an important contribution on the part of many.

The new Book of Standards comprises seven parts instead of six as in 1949. In order to make the volumes less bulky and easier to handle, Bible paper has been used. Some of the compilations are also published on Bible paper as overruns from the Book of Standards.

The committee approved an innovation in make-up of the ASTM BULLETIN whereby certain types of text matter are interspersed with the advertising in the back of the BULLETIN, namely, News Notes on Laboratory Supplies and Testing Equipment, Personals, Necrology, Book Reviews, and similar material.

A special Study Committee was set up to review the format and make-up of our publications. This committee, consisting of L. S. Reid, Chairman, R. C. Adams, and L. W. Wood, has already suggested certain changes in format in connection with the ASTM BULLETIN. It is felt that the changes suggested will make the BULLETIN more attractive, readable, and newsworthy, and hence a more valuable publication. This study committee is now reviewing the format of other Society publications.

For some time the Papers Committee has been very much concerned over the quality of presentation of technical papers at Society meetings. An article on this subject appeared in ASTM BULLETIN No. 181, April, 1952. At the February meeting of the committee the committee gave consideration to the institution of a scheme for recognizing good presentations of papers. This will be tried at the forthcoming Annual Meeting. A subcommittee consisting of R. C. Adams, Chairman, L. E. Gregg, and L. L. Wyman was appointed to administer this plan.

It may be of interest to note that apart from the regular publications, such as *Proceedings* and Book of Standards, 38 publications were issued by the Society during the past year (including 14 compilations). Also, seven reprints of publications have been struck off as listed later in this report.

Two meetings were held by the Administrative Committee on Papers and Publications, one on November 17 and the other on February 20.

RECORD OF PUBLICATIONS ISSUED THIS YEAR

Regular Publications:

1952 Proceedings, 1335 pp, 7700 copies. Available May, 1953.
1952 Book of ASTM Standards
  Part 1, 1602 pp, 15,500 copies.
  Part 2, 1357 pp, 13,250 copies.
  Part 3, 1662 pp, 11,000 copies.
  Part 4, 1182 pp, 9800 copies.
  Part 5, 1281 pp, 10,200 copies.
  Part 6, 1518 pp, 9500 copies.
  Part 7, 1374 pp, 10,000 copies.
1952 Year Book, 632 pp, 7200 copies.
1952 Index to ASTM Standards, 290 pp, 22,500 copies. Available June.
ASTM BULLETIN, 8 issues (May, 1952, to April, 1953), total number of pages 716, average number of copies 10,993.

A statement of the volume of the principal publications for the past four years is given below:

Publications	1949 Pages	1950 Pages	1951 Pages	1952 Pages
Proceedings	1252	1480	1332	1335
Book of ASTM Standards	8396	....	....	9976
Supplements to Book of ASTM Standards	....	2179	1916	....
ASTM Methods of Chemical Analysis of Metals	....	486	....	....
Index to ASTM Standards	280	274	282	300
Year Book	570	588	608	632
Special Technical Publications	2048	1041	1600	2615
ASTM BULLETIN	728	772	716	716
	13 274	6820	6454	15 574

Special Compilations of Standards Published from April 15, 1952, to April 15, 1953:

Specifications for Steel Piping Materials (A-1), 393 pp, 3000 copies.
ASTM Standards on Wire for Electrical Conductors (B-1), 262 pp, 1000 copies.
ASTM Standards on Copper and Copper Alloys (B-5), 556 pp, 1200 copies.
ASTM Standards on Light Metal and Alloys (B-7), 215 pp, 1000 copies.
ASTM Standards on Mineral Aggregates, Concrete, and Nonbituminous Highway Materials (C-9, D-4), 308 pp, 2600 copies.
ASTM Standards on Paint, Varnish, Lacquer, and Related Products (D-1), 804 pp, 2000 copies.
ASTM Standards on Petroleum and Lubricants (D-2), 834 pp, 5900 copies.
ASTM Standards on Electrical Insulating Materials (D-9), 591 pp, 1200 copies.
ASTM Standards on Rubber and Rubber-Like Materials (D-11), 696 pp, 2400 copies.
ASTM Standards on Soaps and Other Detergents (D-12), 156 pp, 1000 copies.
ASTM Standards on Textile Materials (D-13), 676 pp, 2250 copies.
ASTM Standards on Benzene, Toluene, Zylene, Solvent Naptha (D-16), 59 pp, 1000 copies.

Manual on Industrial Water (D-19), 346 pp, 2000 copies.

ASTM Standards on Plastics (D-20), 654 pp, 2000 copies.

Special Compilations in Prospect:

ASTM Standards on Electrical Heating, Resistance, and Related Alloys (B-4), 225 pp, 750 copies.

ASTM Standards on Electrodeposited Metallic Coatings (B-8), 65 pp, 2000 copies.

ASTM Standards on Gaseous Fuels (D-3), 160 pp, 800 copies.

ASTM Standards on Bituminous Constructional Materials (D-4, D-8), 416 pp, 1000 copies.

ASTM Standards on Paper and Paper Products and Shipping Containers (D-6, D-10), 370 pp, 1200 copies.

ASTM Standards on Adhesives (D-14), 96 pp, 1200 copies.

Special Publications Issued from April 15, 1952, to April 15, 1953:

Summary of Proceedings of the Fifty-Fifth Annual Meeting, 36 pp, 7500 copies.

1952 Marburg Lecture, "Non-Destructive Testing," by R. C. McMaster, 78 pp, 4000 copies.

1952 Gillett Lecture, "Man, Metals, and Power," by N. L. Mochel, 48 pp, 3000 copies.

Symposium on Measurement of Consumer Wants (STP 117), 67 pp, 2500 copies.

Symposium on Surface and Subsurface Reconnaissance (STP 122), 234 pp, 2000 copies.

Symposium on Acoustical Materials (STP 123), 40 pp, 1500 copies.

Symposium on Consolidation Testing of Soils (STP 126), 115 pp, 2000 copies.

Symposium on Determination of Elastic Constants (STP 129), 104 pp, 3000 copies.

Symposium on Direct Shear Testing of Soils (STP 131), 93 pp, 2000 copies.

Symposium on Use of Radioisotopes in Soil Mechanics (STP 134), 40 pp, 2000 copies.

Symposium on Insulating Oil (STP 135), 43 pp, 2000 copies.

Symposium on Recent Developments in the Evaluation of Natural Rubber (STP 136), 117 pp, 2000 copies.

Symposium on Fatigue with Emphasis on Statistical Approach (STP 137), 96 pp, 2500 copies.

Symposium on Testing Adhesives for Durability and Permanence (STP 138), 64 pp, 2000 copies.

Symposium on Statistical Methods for the Detergent Laboratory (STP 139), 48 pp, 2000 copies.

Symposium on Metal Powder and Metal Powder Products (STP 140), 90 pp, 3000 copies.

Symposium on Color Differences, 68 pp, 1200 copies.

ASTM-IP Petroleum Measurement Tables, 541 pp, 5000 copies.

ASTM Manual of Engine Test Methods for Rating Fuels, 358 pp, 3000 copies.

Metal Cleaning Bibliography (STP 90-B), 136 pp, 2000 copies.

Bibliography on Electrical Contacts (STP 56-G), 266 pp, 2250 copies.

Fifty Year Index, 215 pp, 5000 copies.

Bibliography of Fatigue References (STP 9-C), 24 pp, 275 copies (mimeographed).

Bibliography on Soil Dynamics (STP 146), 9 pp, 200 copies (mimeographed).

Manual on Engine Antifreezes (STP 120), 20 pp, 4000 copies. (Subsequently reprinted 6000 copies.)

Special Publications in Prospect:

Symposium on Strength and Ductility of Metals at Elevated Temperatures with Particular Reference to Effects of Notches and Metallurgical Changes (STP 128), 255 pp, 3000 copies.

Symposium on Continuous Analysis of Industrial Water and Industrial Waste Water (STP 130), 58 pp, 2000 copies.

Symposium on Plastics Testing (STP 132), 81 pp, 2000 copies.

Symposium on Conditioning and Weathering (STP 133), 104 pp, 2500 copies.

Symposium on Tin, (STP 141), 100 pp, 2000 copies.

Symposium on Exchange Phenomena in Soils (STP 142), 81 pp, 2000 copies.

Symposium on Light Microscopy (STP 143), 146 pp, 2500 copies.

Symposium on Fretting Corrosion (STP 144), 96 pp, 2000 copies.

Symposium on Non-Destructive Testing (STP 145), 100 pp, 2500 copies.

Symposium on Paint (STP 147), 70 pp, 2500 copies.

Symposium on Chemical Analysis of Inorganic Solids by Means of the Mass Spectrometer (STP 149), 50 pp, 2000 copies.

Symposium on Soaps and Other Detergents, 60 pp, 2000 copies.

Manual on Measurement and Sampling of Petroleum and Petroleum Products, 182 pp, 2000 copies.

Proposed Procedures for Emission Spectrochemical Analysis, 250 pp, 2000 copies.

Index to the Literature on Spectrochemical Analysis, 200 pp, 2000 copies.

Bibliography on Speed of Testing, 20 pp, 500 copies (mimeographed).
Bibliographical Abstracts and Methods for Analysis of Synthetic Detergents, 25 pp, 2000 copies.

Reprints:

Symposium on New Methods for Particle Size Determination in the Subsieve Range (1941), 111 pp, 800 copies.
1951 Marburg Lecture, "Corrosion Testing," by F. L. LaQue, 92 pp, 750 copies.
Symposium on Usefulness and Limitations of Samples (1948), 48 pp, 1000 copies.
Index to the Literature on Spectrochemical Analysis (1947), 184 pp, 500 copies.
Significance of Tests of Petroleum Products (1934), 80 pp, 1500 copies.
Radiography of Steel Castings, 25 sets each of X-ray and Gamma-ray, 32 negatives in each.
Cotton Yarn Appearance Standards, Series 1-110; 2-143; 3-195; 4-154; 5-117.

CURRENT ACTIVITIES

A very full program is in prospect for the 1953 Annual Meeting, with a number of symposiums scheduled as follows:

Symposium on Metallic Materials at Low Temperatures.
Symposium on Techniques for Electron Metallography.
Symposium on X-ray Spectrographic Analysis.
Symposium on Lateral Pile Load Tests.
Symposium on Soil Dynamics.
Symposium on Porcelain Enamels and Ceramic Coatings as Engineering Materials.
Symposium on Radioactivity in ASTM Work.
Session on Significance of Tests of Concrete.

In addition to the above, there are a number of other technical papers which will be presented at the Annual Meeting. A number of papers are being offered to the Society for publication but not presentation at a meeting. These for the most part will be published currently in the ASTM BULLETIN or in the *Proceedings*.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The last meeting of the Administrative Committee on Simulated Service Testing was held at Society Headquarters on November 24, 1952.

The agenda covered the following items:

1. As an aid to those not thoroughly familiar with the performance of metals at high temperatures, it would seem appropriate to have available a source of information which would give some idea as to the methods and the relative importance of tests for the evaluation of metals for various high-temperature applications. This was considered most properly an activity which should fall within the scope of the Joint Committee on Effect of Temperature on Metals and will be referred to that group.

2. The problem of machinability, with suitable standards is recurrent and unanswered. However, it is understood that a group in Technical Committee K of ASTM Committee D-2 is studying cutting fluids in lathe turning tests and the activities of this group may lead to a symposium on this subject. It was decided to contact other societies such as the Society of Automotive Engineers, The American Society of Mechanical Engineers, and The American Society of Tool Engineers, to learn the extent of their interest in the subject and to gather constructive information that might be used later for organizing a symposium.

3. The increasing importance of shot peening has emphasized the necessity for adequate tests for satisfactory peening. The committee advised that a new SAE Division 20 is now working on this problem and hopes to establish recommendations.

It was brought out in the discussion that, given a satisfactory intensity (as determined by an arc-height Almen test strip), the major problem was one of coverage, wherein an 80 per cent shot peen coverage (estimated by visual examination) is considered good.

4. In connection with bend testing—a subject of continued and active interest in the Administrative Committee—it is encouraging to note that Committee E-1 now has a new task group to study such tests. The many suggested tests, correlation of tests with application, value of bend tests now on the books, relation of bend tests to spring-back, formability, etc., which have long been discussed in the committee will be passed on to this group.

5. Other items which were considered include Fretting Corrosion, Humidity Testing, Gaskets, Radiographic Standards, Macroetching, and Decarburization.

It is anticipated that a paper on the philosophy of simulated service testing will soon be ready for presentation to the Society by one of the committee members.

Respectfully submitted on behalf of the Committee,

L. L. WYMAN,
Chairman.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

The most noteworthy district activity during the past year was the formation in March of the new Southwest District. This new district encompasses the areas of Texas, Oklahoma, Louisiana and the part of Arkansas not covered by the St. Louis District. The Chairman of this new District is Milton Holmberg, Houston, Tex., assisted by Vice-Chairmen R. C. Alden, Bartlesville, Okla.; A. W. Eatman, Austin, Tex.; P. L. DeVerter, Baytown, Tex.; and Edwin Joyce, Dallas, Tex.; and Secretary J. B. Baird, Houston, Tex.

The New York District Council was the nucleus of The General Committee on Arrangements which acted as host for the Fiftieth Anniversary Meeting of the Society, planning the ladies entertainment, handling details of the annual dinner, and underwriting and sponsoring the entertainment program. In connection with the Annual Meeting, the customary breakfast of the Administrative Committee on District Activities was held with the officers of the various districts as guests of the Society.

Meetings:

The thirteen previously established districts functioned smoothly throughout the year, holding 21 meetings including the Detroit District Meeting held in conjunction with the ASTM Spring Meeting. Dr. Kenneth McFarland, the speaker at this meeting, addressed a group of several hundred members and their wives on "Fathoming the Fifties." The only districts which did not hold meetings during the June, 1952–June, 1953 year were Cleveland, Chicago, and Western New York-Ontario.

Two meetings of note were held outside the province of ASTM Districts. These included a meeting in Richland, Washington in conjunction with the American Society of Metals and the American Institute of Chemical Engineers and a meeting in Birmingham, Ala. in conjunction with the American Chemical Society, the ASM, and ASTM Committee A-1 on Steel. Both of these meetings were highly successful and suggest areas for future development.

President's Addresses:

President Maxwell toured the country speaking at Richland, Wash.; Birmingham, Ala.; and Northern California, Southern California, Southwest, New England, Philadelphia, St. Louis, and Pittsburgh Districts. His talk on "Chemical Developments and Engineering Materials" was well received, and special efforts were made to publicize his talk among the various students in these areas. Dr. Maxwell also spoke in Richmond, Va., at the Washington, D. C., District Meeting on the subject of "Research and Standards."

Many of the most successful district meetings have been held in conjunction with other societies such as American Society for Quality Control, Society for Experimental Stress Analysis, Society for Non-Destructive Testing, Inc., Engineers' Club of St. Louis, AIChE, ASM and ACS. The details of the district meetings are listed in the table of meetings included in the body of the report.

In the matter of membership, there has been a considerable number of personal and direct contacts. In addition, the Washington, D. C., District made a special mailing to all of its members asking for suggestions concerning individuals for Society membership.

Respectfully submitted on behalf of the committee,

C. M. Gambrill,
Chairman

REPORT OF COMMITTEE A-1

ON

STEEL*

While there were a number of important developments in the work of Committee A-1 during the past year, by far the most outstanding was the proposed Tentative Methods and Definitions for the Mechanical Testing of Steel Products.[1] Although work on this document has been under way since 1950, it was not expected to reach completion before 1955. However, since the 1952 Annual Meeting many of the members of Committee A-1 have spent extraordinary amounts of time on the project because of its importance to the steel industry. The result has been a coordination of many divergent views on the production testing of steel products.

In addition to these activities, forty-six recommendations have been approved by the Administrative Committee on Standards. These are outlined in detail in another section of this report.

One of the important actions taken was the publication of an appendix to the Tentative Method of Macroetch Testing and Inspection of Steel Forgings (A 317 – 52 T),[2] illustrating the various types of defects which can be revealed by macroetching steel forgings. Still another was the publication of revisions of the Tentative Specifications for Carbon Steel and Alloy Steel Blooms, Billets, and Slabs for Forgings (A 273 – 52 T and A 274 – 52 T),[2] which should result in increased usage of these two specifications. Revisions in these three standards have been under consideration for a number of years.

With the publication late in 1952 of the Tentative Specifications for Cold-Finished and Heat-Treated Alloy Steel Bars (A 364 – 52 T),[2] Committee A-1 has a complete coverage by its specifications of the steel bar field. The complete list includes carbon steel bars which can be ordered either to chemical composition or mechanical properties, stress-relief-annealed steel bars, alloy steel bars for nitriding, and alloy steel bars to hardenability requirements. Hot-rolled, cold-finished, and heat-treated carbon and alloy steel bars are covered.

Widely used Tentative Specifications A 158, A 206, A 280, and A 315 for alloy steel pipe for high-temperature service were discontinued since these grades of steel are now present in the Tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 52a T).[2]

Committee A-1 departed from its usual practice of meeting in either Pittsburgh, Philadelphia, or Cleveland in late January or early February, and visited Birmingham, Ala. for a three-day meeting in February, 1953. The total attendance surpassed the mid-winter meetings in 1952 and 1951 in Philadelphia and Cleveland, respectively. Many actions on standards were taken at this meeting, as reflected in the Appendix to this report.[3]

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 1.

[2] 1952 Book of ASTM Standards, Part 1.

[3] See p. 87.

Personnel.—As a result of personnel changes during the past year, the committee now consists of 289 voting members, including 131 producers, 116 consumers, and 42 general interest members.

Because of retirement, Jonathan Jones has resigned as vice-chairman of Subcommittee II on Structural Steel and has been replaced by J. F. Willis of the Connecticut State Highway Dept. Both Subcommittee II and Committee A-1 regret the loss of Mr. Jones who has very ably represented the consumers of structural steel for many years.

Another very able consumer leader was lost in Subcommittee XI on Steel for Boilers and Other Pressure Vessels. H. G. Miller, Chairman of Subcommittee XI retired from his position as Mechanical Engineer for the Chicago, Milwaukee, St. Paul & Pacific Railroad Co. R. W. Mooty, Tennessee Coal and Iron Div., U. S. Steel Corp., has been named as successor to Mr. Miller.

To fill the position of vice-chairman of Subcommittee XXII on Steel for High Temperatures and Subatmospheric Temperatures left vacant in 1952 when C. A. Kelting was named chairman, W. B. Hoyt of the M. W. Kellogg Co. has been appointed.

R. B. Hooper has resigned as chairman of the Special Subcommittee on Hardenability and H. H. ZurBurg of the Chrysler Corp. has accepted the appointment as successor.

The committee records with regret the death during the past year of several present and former well-known members of Committee A-1, including F. C. Fyke, R. S. Johnson, L. B. Jones, W. W. Scott, Jr., B. J. Schmid, P. H. Smith, F. M. Waring, and G. B. Waterhouse. Mr. Fyke had represented the Standard Oil Development Co. on Committee A-1 since 1930, and likewise Mr. Scott had represented the Laclede Steel Co. since 1925. P. H. Smith for many years had represented the Chicago, Burlington, & Quincy Railroad Co. and was quite active in subcommittee work. Mr. Waterhouse, Emeritus Professor of Metallurgy, Massachusetts Institute of Technology, was an old-time member of the committee, as was Mr. Johnson, formerly Director of Research for John A. Roebling Sons Co. Mr. Schmid, although a relatively new member of the committee, representing the Pacific Gas and Electric Co., San Francisco, Calif., was one of the first West Coast members of Committee A-1.

Many of the present members remember Mr. Waring, who was Engineer of Tests for the Pennsylvania Railroad during his active interest in Committee A-1. Mr. Waring was chairman of the committee from 1920 to 1926 and was elected an honorary member of the committee. L. B. Jones, also Engineer of Tests for the Pennsylvania Railroad before his retirement in 1948, gave unstintingly of his knowledge and sound judgment in the operations of Committee A-1 for many years. Mr. Jones was also elected to honorary membership in Committee A-1 in 1950.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On June 13, 1952, the Administrative Committee on Standards accepted the recommendation of Committee A-1 that the Tentative Method of Macroetch Testing and Inspection of Steel Forgings (A 317 – 48 T) be revised. The revised method appears in the 1952 Book of ASTM Standards, Part 1.

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Steel for Bridges and Buildings (A 7 – 52 T),

Structural Nickel Steel (A 8 – 52 T),
Boiler and Firebox Steel for Locomotives (A 30 – 52 T),
Structural Silicon Steel (A 94 – 52 T),
Structural Steel for Locomotives and Cars (A 113 – 52 T),
Open-Hearth Iron Plates of Flange Quality (A 129 – 50 T),
Structural Steel for Ships (A 131 – 52 T),
Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 52 T),
Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 52 T),
Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 52 T),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 52 T),
High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 52 T),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 52 T),
Low-Alloy Structural Steel (A 242 – 50 T),
Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 – 47 T),
Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 274 – 44 T),
Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 283 – 52 T),
Low and Intermediate Tensile Strength Carbon-Silicon Steel Plates of Structural Quality (A 284 – 52 T),
Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (Plates 2 in. and Under in Thickness) (A 285 – 52 T),
High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 – 52 T),
Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 52 T),
Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 52 T), and
Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Washers (A 325 – 51 T).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the revised tentative specifications appear in the 1952 Book of ASTM Standards, Part 1.

The following recommendations submitted by Committee A-1 were accepted by the Administrative Committee on Standards on October 21, 1952:

Tentative Specifications for:

Cold Finished Heat Treated Alloy Steel Bars (A 364 – 52 T).

Revision of Tentative Specifications for:

General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 – 52 T),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 51 T),
Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 52 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 51 T),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 51 T),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 51 T),
Seamless Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 52 T),
Factory-Made Wrought Carbon Steel and Ferritic Alloy-Steel Welding Fittings (A 234 – 52 T),
Alloy-Steel Bars to End-Quench Hardenability Requirements (A 304 – 52 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 51 T),
Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 52 T), and
Alloy Steel Bars for Nitriding (A 355 – 52 T).

Tentative Revision of Standard Specifications for:

Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 46), and
Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 49).

Withdrawal of Tentative Specifications for:

Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 52 T),
Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 52 T),
Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 52 T),
Seamless 1 per cent Chromium, 0.5 per cent

Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 315 - 52 T).

The new and revised tentative specifications and the tentative revisions of the standard specifications appear in the 1952 Book of ASTM Standards, Part 1.

On December 12, 1952, the Standards Committee approved revisions in the following specifications which appear in the 1952 Book of ASTM Standards, Part 1:

Revision of Tentative Specifications for:

Billet-Steel Bars for Concrete Reinforcement (A 15 - 50 T),
Rail-Steel Bars for Concrete Reinforcement (A 16 - 50 T), and
Axle-Steel Bars for Concrete Reinforcement (A 160 - 50 T).

RECOMMENDATIONS AFFECTING STANDARDS

Committee A-1 is presenting for publication 3 new tentatives and a tentative revision to 1 standard, is recommending the revision of 19 tentatives and 4 standards, and the adoption as standard of 3 tentatives and 1 tentative revision of a standard. An editorial revision of 1 specification is also being recommended.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Only the most salient of the numerous current subcommittee activities will be covered in the following paragraphs.

The Standards Division of the General Services Administration has requested Subcommittee II to develop a specification for a weldable grade of structural steel, and this document is being prepared.

Subcommittee IV has two chromium-silicon steel wire specifications in their final stages. One of these specifications covers valve spring quality wire and the other covers commercial quality wire.

A specification covering carbon and alloy steel forgings for light weight pressure vessel shells such as gas cylinders is being given final consideration in Subcommittee VI and will very probably be published late in 1953.

Because of the objections of the ASME Boiler Code Committee to Specifications A 155 for Electric-Fusion-Welded Steel Pipe for High-Temperature Service,[2] Subcommittee XXII has decided to eliminate class 2 from the specifications and to reword the specifications to comply with the requirements of Sections I and VIII of the Boiler Construction Code. The class 2 material will be covered by new specifications. New specifications for seamless and welded austenitic steel pipe for high-temperature service are being written, since difficulty is encountered in obtaining pipe under Specification A 312 for this type of service. Other new specifications for seamless ferritic alloy steel forged and bored pipe for high-temperature service are being developed.

This report has been submitted to letter ballot of the committee, which consists of 289 members; 214 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

J. S. WORTH,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Withdrawal of Emergency Alternates (Relating to Molybdenum) in Specifications for:

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (EA – A 182),
Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (EA – A 217),
Seamless Ferritic Alloy-Steel Pipe for High-Temperature Service (EA – A 335),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (EA – A 352),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (EA – A 161),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (EA – A 199),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (EA – A 200),
Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (EA – A 209),
Seamless Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (EA – A 213), and
Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (EA – A 250).

These recommendations were accepted by the Standards Committee on September 9, 1953.

The following recommendations of Committee A-1 were accepted by the Standards Committee on December 16, 1953:

Tentative Specifications for:

Ferritic Alloy-Steel Forged and Bored Pipe for High-Temperature Service (A 369 – 53 T), and
Carbon and Alloy-Steel Forgings for Pressure Vessel Shells (A 372 – 53 T).

Revision of Tentative Specifications for:

General Requirements for Structural Steel (A 6 – 52a T),
Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 51 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 53 T),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 52 T),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 53 T),
Seamless Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes (A 213–52 T),
Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 51 T),
Carbon Steel Forgings for Locomotives and Cars (A 236 – 52 T),
Low-Alloy Structural Steel (A 242 – 52 T),
Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger and Condenser Tubes (A 249 – 53 T),
Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Washers (A 325 – 52 T),
Seamless Ferritic Alloy-Steel Pipe for High-Temperature Service (A 335 – 52a T),
Low-Carbon High-Nickel Steel Plate for Pressure Vessels (A 353 – 52 T), and
Quenched and Tempered Alloy-Steel Bolts and Studs with Suitable Nuts (A 354 – 52 T).

Withdrawal of Tentative Methods for:

Mechanical Testing of Structural Steel (A 359 – 52 T).

Withdrawal of Tentative Methods and Definitions for:

Mechanical Testing of Steel Bars (A 330 – 51 T).

Withdrawal of Emergency Alternate Provisions in Specifications for:

Heat-Treated Alloy-Steel Bars (EA – A 286),
Alloy-Steel Bars to End-Quench Hardenability Requirements (EA – A 304),
Hot-Rolled Alloy-Steel Bars (EA – A 322), and
Cold-Finished Alloy-Steel Bars (EA – A 331).

The new and revised tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 1.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix certain recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in the 1952 Book of ASTM Standards, Part 1.

NEW TENTATIVES

The committee recommends that the following three new tentatives, as appended hereto, be accepted for publication as tentative:

Tentative Methods and Definitions for the Mechanical Testing of Steel Products:[1]

These methods and definitions have been developed to furnish the steel industry, producers and consumers alike, with standardized procedures for the production testing of steel products. All other similar testing methods published by the Society at the present are cover-all methods for ferrous and nonferrous metals, and in the opinion of Committee A-1 reflect laboratory or research procedures rather than production procedures.

Tentative Specifications for Cold Rolled Carbon Steel Deep Drawing Sheet, Special Killed for Miscellaneous Drawn or Severely Formed Parts:[1]

These specifications cover deep drawing sheet in coils or cut lengths, primarily intended for drawn or severely deformed parts requiring good surface finish. The material is also for use where long storing periods are involved and where roller leveling facilities are not available for prevention of stretcher straining. The specifications are not applicable where material is ordered within a guaranteed breakage allowance.

Tentative Specifications for Cold-Rolled Carbon Steel Sheet, Commerical Quality:[1]

These specifications cover cold-rolled carbon steel sheet in coils and cut lengths. This material is used primarily for bending and moderate forming.

REVISIONS OF TENTATIVES

The committee recommends revisions in the following 19 tentatives:

Tentative Specification for Steel for Bridges and Building (A 7 – 52 T):

Table III.—Revise Footnote *a* to read "The upper limit of 72,000 psi may be increased by 3000 psi for all thicknesses of shapes and for other material over 1½ in. in thickness."

Tentative Specifications for Quenched Carbon-Steel Joint Bars (A 49 – 51 T):

Section 9.—In Paragraphs (*a*) and (*b*) change the requirement for the diameter of the pin for the bend test from the present "equal to three times . . ." to read "not greater than three times . . . ."

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[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 1.

Tentative Specification for Structural Steel for Ships (A 131 – 52 T):

Section 4 (b).—Revise to read as follows:

> (*b*) Plates over $\frac{1}{2}$ to 1 in., inclusive, in thickness shall conform to the requirements for grade B prescribed in Table I. Plates over 1 to $1\frac{3}{8}$ in., inclusive, in thickness shall conform to the requirements of grade C prescribed in Table I. Plates over $1\frac{3}{8}$ in. in thickness shall conform to the requirements for grade C prescribed in Table I except that, if intended for use in important structural parts, they may be required to be produced to special specifications.

TABLE I.—CHEMICAL REQUIREMENTS FOR FREE-MACHINING 18 CHROMIUM-8 NICKEL STEEL (AISI TYPE 303).

	With Added Sulfur		With Added Selenium	
	Range	Check Variation, Over or Under	Range	Check Variation, Over or Under
Carbon, per cent	0.15 max	0.01 over	0.15 max	0.01 over
Manganese, per cent	2.00 max	0.04 over	2.00 max	0.04 over
Phosphorus, per cent	0.06 max	0.010 over	0.17 max	0.010 over
Sulfur, per cent	0.18 to 0.35	0.020	0.10 max	0.010 over
Silicon, per cent	1.00 max	0.05 over	1.00 max	0.05 over
Nickel, per cent	8.00 to 10.00	0.10	8.00 to 10.00	0.10
Chromium, per cent	17.00 to 19.00	0.20	17.00 to 19.00	0.20
Selenium, per cent	....	....	0.15 to 0.35	0.03

Tentative Specification for Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 52 T):

Table III.—In the column for "Calculated Weight per Foot" for all sizes of tubes change the permissible over variation from the present "3.5" to read "5" per cent.

Add a new footnote reference after the heading for this column to read "The calculated weight per foot shall be determined by the following formula:

$$W = 10.68 \left(D - \frac{t}{0.875} \right) \frac{t}{0.875}$$

where:

W = weight in pounds per foot,
D = outside diameter in inches, and
t = specified minimum wall thickness in inches.

Tentative Specifications for Electric-Resistance Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 51 T):

Section 7(a).—Change the requirement for width of flange for types A and B tubes over $2\frac{1}{2}$ to $3\frac{3}{4}$ in. in outside diameter from the present "$\frac{3}{8}$ in." to read "$12\frac{1}{2}$ per cent of outside diameter."

Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 52a T):

Table I.—For grade F 12 (1 per cent chromium, 0.5 per cent molybdenum) change the requirement for chromium content from "0.80 to 1.10" to read "0.85 to 1.20" per cent.

Tentative Specifications for Heat-Treated Carbon and Alloy Steel Track Bolts and Nuts (A 183 – 51 T):

Title.—Change to read "Tentative Specifications for Heat-Treated Carbon and Alloy Steel Track Bolts and Carbon Steel Nuts."

Section 9.—Renumber as Section 7.

Section 7.—Renumber as Section 8 and in Paragraph (*a*) change the requirement for the diameter of the pin for the bend test from the present "equal to the diameter of the bolt" to read "not greater than the diameter of the bolt."

Section 8.—Renumber as Section 9. In footnote *a* to the table in this section delete the last sentence and add the following as a second paragraph: "The above table is predicated on the minimum thickness of a medium-carbon steel nut being equal to the nominal bolt diameter and the minimum thickness of a low-carbon steel nut being ⅛ in. larger than the nominal bolt diameter."

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 52 T):

Table I.—Change the chemical requirements for grade B8F to read as shown in the accompanying Table I.

Tentative Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 52 T):

Table III.—Revise as indicated above for Table III in Specifications A 161.

Tentative Specification for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 47 T):

Section 10.—In Paragraph (*a*) revise so read "Grade WCA castings ordered under these specifications shall be satisfactorily tested after machining to such pressures as . . . "

In Paragraph (*b*) revise the first sentence to read "Grade WCB castings shall be individually tested after machining to the hydrostatic . . . "

Add a new Paragraph (*c*) to read as follows:

> (*c*) It is realized that the foundry cannot perform the required pressure testing at the high pressure required on unmachined castings. Castings ordered in the rough state for final machining by the purchaser may be hydrostatically tested by the manufacturer at pressures to be agreed upon with the purchaser. However, the foundry is responsible as required under Section 20 (*b*) for the satisfactory performance of the castings under the final test required in Section 10 (*a*) or (*b*).

Tentative Specification for Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 49 T):

Section 1(*b*).—Change "seven grades of ferritic alloy steels" to read "eight grades of ferritic alloy steels."

Make this same change in Note 3.

Table I.—Add a new grade of steel with the following chemical requirements:

Carbon, max, per cent	0.18
Manganeses, per cent	0.40 to 0.70
Phosphorus, max, per cent	0.05
Sulfur, max, per cent	0.06
Silicon, max, per cent	0.60
Chromium, per cent	1.0 to 1.5
Molybdenum, per cent	0.40 tg 0.60
Vanadium, per cent	0.15 to 0.25

Table II.—Add the new grade with the same requirements for unspecified alloying elements as grade WC6.

Table III.—Add the new grade with the same tensile requirements as WC4, WC5, WC6, and WC9.

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes For High-Pressure Service (A 226 – 51 T):

Section 7(*a*).—Revise the second sentence as follows by the addition of the italicized fraction and the deletion of the fraction in brackets: "This flange, as measured from the outside of the tube, shall not be less than ⅛ in. nor more than [⅜ in.] *1/2 in.*"

In the table change the width of flange for tubes over 2½ to 3¾ in. in outside diameter from the present "⅜ in." to read "12½ per cent of outside diameter."

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger and Condenser Tubes (A 249 – 52 T):

Section 8.—Revise the second sentence

as follows by the addition of the italicized fraction and the deletion of the fraction in brackets: "This flange, as measured from the outside of the tube, shall not be less than $\frac{1}{8}$ in. or more than [$\frac{3}{8}$ in.] *3/4 in.*"

Tentative Specifications for Electric-Resistance-Welded Carbon Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 51 T):

Section 8.—Revise the second and third sentences by the addition of the italicized numbers and the deletion of the numbers in brackets: "This flange, as measured from the outside of the tube, shall be not less than $\frac{1}{8}$ in. or more than [$\frac{3}{8}$ in.] *1/2 in.* Within these limits the width of the flange shall not be less than [15 per cent] *10 per cent* of the outside diameter of the tube."

Tentative Specification for Steel Plates for Pressure Vessels for Service at Low Temperature (A 300 – 52 T):

Table I.—Include as class 4 the 9 per cent nickel steel covered by the Tentative Specification for Low-Carbon, High-Nickel Steel Plate for Pressure Vessels (ASTM Designation: A 353).[2]

Section 4(d).—Revise the last sentence to read "If the purchase order bears the notation 'stress relieve specimens,' the test pieces shall receive a subsequent stress relieving treatment at 1100 to 1200 F, prior to preparation of the impact specimens, except for class 4 which shall be stress relieved at a temperature of 1025 to 1085 F."

Tentative Specifications for Minimum Requirements for the Deformation of Deformed Steel Bars for Concrete Reinforcement (A 305 – 50 T):

Section 4(a).—Add the following sentence "A measured length of the bar specimen shall be considered the distance from a point on a deformation to a corresponding point on any other deformation on the same side of the bar."

Tentative Specifications for Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 52 T):

Section 5(d).—Revise to read as follows:

> (*d*) All nuts, except jam, slotted and castellated nuts shall meet the proof load specified in Table I. When equipment of sufficient capacity is not available for test, nuts over $1\frac{1}{8}$ in. in diameter shall be accepted on the basis of a minimum hardness of 104 Brinell.

Table I.—Change the heading for the last column from "Stripping Load" to read "Proof Load."

Section 6(a).—In the second and third sentences change the gage length from the present 3 threads to be 6 threads.

Tentative Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 52 T):

Table I.—Change the chemical requirements for grade L8F to read as shown in the accompanying Table I.

Tentative Specifications for Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 – 52 T):

Section 3(a).—Change the minimum tempering temperature from the present "800 F" to read "850 F."

Section 5.—In Paragraphs (*b*), (*d*) (*1*), and (*d*) (*3*) change the gage length from one diameter to six threads.

ADOPTION OF TENTATIVES AS STANDARDS

The committee recommends that the following two specifications be approved for reference to letter ballot of the Society for adoption as standard without revision:

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[2] 1952 Book of ASTM Standards, Part 1.

Tentative Specifications for:
Electric-Fusion (Arc)-Welded Steel Plate Pipe (Sizes 16 in. and over) (A 134 – 51 T), and
Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and Over) (A 139 – 51 T).

The committee recommends that the following tentative be approved for reference to letter ballot of the Society for adoption as standard with revision as indicated:

Tentative Specifications for Open-Hearth Carbon-Steel Rails (A 1 – 49 T):

Section 7.—Revise to read as follows:

> Separate analyses shall be made from ladle tests representing one of the first three and one of the last three applied full ingots of the heat to determine the percentage of carbon, manganese, phosphorus, sulfur, and silicon. Determinations may be made chemically or spectrographically. The average analysis of the ladle tests shall conform to the requirements prescribed in Table I.

TENTATIVE REVISION OF STANDARD

The committee recommends a revision of the following standard for publication as tentative:

Standard Specifications for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 37):

The tentative revision, as appended to this report,[3] is intended to replace Standard Specifications A 185 when adopted. It includes the addition of definite weld tests and procedures to limit the weld strength in shear for fabric to a minimum of 35,000 psi, based on the area of the longitudinal wire.

ADOPTION OF TENTATIVE REVISION OF STANDARD

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 52):

The committee recommends that the present tentative revision of Section 12(*a*) reducing the number of tension tests required from 2 per cent to 1 per cent be adopted as standard and incorporated in these specifications.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following four standards be revised as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these may be referred to letter ballot of the Society.

Standard Specifications for High-Carbon Steel Joint Bars (A 5 – 48):

Section 1.—Add a sentence to read "They may be used for the joint bars of insulated joints."

Standard Specifications for Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 51):

Section 1.—Delete reference to grade 0 nuts here and throughout the specifications.

Table I.—In addition to deleting the requirements for grade 0, revise the chemical requirements for grade 8F to read as shown in the accompanying Table I.

Section 8.—Revise Paragraphs (*a*) and (*b*) to read as follows:

> (*a*) Samples of each grade of nuts shall show the hardness prescribed in Table II in the finished condition.
>
> (*b*) In addition, samples of grades 1, 2, 2H, 3 and 4 nuts shall show the hardness prescribed in Table II after they have been subjected for 24 hr to a temperature of 850 F (455 C) for grade 1, 1000 F (540 C) for grades 2 and 2H, and 1100 F (590 C) for grades 3 and 4, and then cooled slowly.

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[3] The tentative revision was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 1.

Section 10.—Add another sentence to read "This test will not be required for nuts which can not be tested on a machine of 60,000 lb capacity."

Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 47):

Section 11(a).—Revise to read as follows by deleting the words in brackets and adding the italicized words: "From each group of 100 finished tubes or fraction thereof, (two tubes) *one tube* shall be selected at random for the tension test specified in Section 6."

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 52):

Table II.—Revise as indicated above for Table III in Tentative Specifications A 161.

EDITORIAL CHANGE

The committee recommends the following editorial change:

Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 – 48):

Appendix II.—Delete the tables of wheel design and substitute the following notes: "The dimensions of standard steel wheel designs are published by the American Transit Association, 292 Madison Ave., New York 17, N. Y."

REPORT OF COMMITTEE A-2

ON

WROUGHT IRON

Committee A-2 continues to be active following its reorganization in 1951. At the June 30, 1953, meeting, the following items were proposed to be considered during 1953:

Standard Specifications for Wrought Iron Rivets and Rivet Rounds (A 152 – 39).—Standard Specifications A 152 should be revised to permit only double refined wrought iron to be used for the manufacture of wrought iron rivets.

Standard Specifications for Refined Iron Bars (A 41 – 36).—These specifications should be withdrawn since Specifications A 189 – 42 and A 207 – 39 completely cover the subject.

Standard Specifications for Wrought Iron Rolled or Forged Blooms and Forgings (A 73 – 39).—In view of the action on Specifications A 41 it is proposed to delete class B from Specifications A 73.

Proposed Specifications for Cold-Drawn Wrought Iron Heat Exchanger and Condenser Tubes.—This product is being manufactured and used extensively enough that ASTM Specifications would serve a useful purpose. Subcommittee I will prepare a draft for consideration of the committee. Cooperation of Committee A-2 with the British Wrought Iron Association was discussed with W. S. Gierszynski, Thomas Wolmsley & Sons, Ltd., Bolton, England, who was present at the meeting of the committee.

Respectfully submitted on behalf of the committee,

A. D. Morris,
Chairman.

L. S. Crane,
Secretary.

REPORT OF COMMITTEE A-3
ON
CAST IRON*

Committee A-3 on Cast Iron held two meetings during the past year: one on June 26, 1952, in conjunction with the Annual Meeting of the Society, and the other in Detroit, Mich., on March 4, 1953.

At the present time, the committee consists of 115 members, of whom 40 are classified as producers, 47 as consumers, and 28 as general interest members.

Committee A-3 is honored by having one of its most active members and a past chairman, James T. MacKenzie, selected by the Society to receive a 1953 ASTM Award of Merit.

NEW TENTATIVE

The committee recommends that the Methods of Chill Testing of Cast Iron as appended[1] to this report be accepted for publication as tentative.

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Automotive Gray Iron Castings (**A 159 - 49T**)[2] be revised as follows:

Section 2(b).—At the end of the first sentence, add the words "suitably ground for the test."

Section 3.—Revise Paragraph (*a*) to read "The transverse test shall be made as described in the Specifications for Gray Iron Castings (ASTM Designation: A 48)."

Revise Paragraph (*b*) to read as follows:

> Values shall conform to the requirements prescribed in Table I. In case the size of the transverse bar is not specified, the 1.2-in. diameter bar shall be used.

Table II.—Revise the first sentence of Footnote (*d*) to read "Heavy duty drums and clutch plates where both strength and resistance to heat checking are definite requirements. . . . ."

Section 4.—Delete the Note and add the following Paragraph (*c*):

> In such instances, the physical properties as represented by test bars should be in agreement with the specifications as supplied by the purchaser. The minimum values shall be established by joint agreement. Tension tests taken from castings may not agree with tests taken from test bars.

Appendix, Section A1.—For emphasis print this paragraph in bold face italics as follows:

"The data in this appendix are furnished as information only and do not constitute a part of these specifications as given in Table I."

Appendix, Table III.—Delete sulfur contents in the fifth column and insert 0.15 per cent for all five grades.

Appendix, Section A4, Paragraph No. 120.—Delete the words "cylinder sleeves."

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following tentative specifications be adopted as standard without revision:

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 1.

[2] 1952 Book of ASTM Standards, Part 1.

Specifications for Gray Iron Castings for Pressure Containing Parts for Temperatures up to 650 F (**A 278 - 51 T**),[2] and

Specification for Gray Iron Castings for Elevated Temperatures for Non-Pressure Containing Parts (**A 319 - 48 T**).[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued without revision:

Specifications for Foundry Pig Iron (A 43 - 49 T)
Methods of Impact Testing of Cast Iron (A 327 - 50 T), and
Specifications for Nodular Iron Castings (A 339 - 51 T).

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends that the Specifications for Culvert Pipe (A 142 - 38) be continued without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VI on General Castings (H. W. Lownie, chairman) is considering revision of Specifications A 48 - 48 to bring it up to date.

Subcommittee VII on Microstructure of Cast Iron (Robert Lindsay, chairman) is preparing a graphite classification chart for forms of graphite found in nodular iron.

Subcommittee X on Nomenclature and Definitions (Alfred Boyles, chairman) is now undergoing reorganization.

Subcommittee XVI on Heat Treatment of Cast Iron (V. T. Malcolm, chairman) is considering the advisability of raising the stress relieving temperature specified in Specifications A 278 - 51 T.

Subcommittee XVIII on Automotive Castings (V. A. Crosby, chairman) has recommended revisions of Specifications A 159 - 49 T referred to previously in this report.

Subcommittee XIX on Chilled and White Iron Castings (J. J. Marsalka, chairman) had its Specifications for Chilled and White Iron Castings accepted by the Society and designated as A 360 - 52 T. A recommended method for correctly measuring depth of chill is being considered.

Subcommittee XXI on Pressure Pipe (T. H. Wiggin, chairman) is reviewing the specifications A21.2, A21.3, A21.6, A21.7, A21.8, A21.9, A21.10, and A21.11 prepared by ASA Sectional Committee A21 for the purpose of recommending suitable specifications for publication by the ASTM.

Subcommittee XXIV on Nodular Iron (T. E. Eagan, chairman) is considering including a normalized grade of nodular iron with a higher tensile strength than covered by present specifications. The matter of selecting a more appropriate name for "nodular" iron has also been discussed.

Subcommittee XXV on Methods of Chill Testing Cast Iron (D. E. Krause, chairman) submitted the Methods of Chill Testing Cast Iron appended hereto[1] for publication as tentative.

This report has been submitted to letter ballot of the committee, which consists of 118 voting members; 75 members returned their ballots, of whom 72 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. S. VANICK,
Chairman.

D. E. KRAUSE,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held meetings on June 25, 1952 at New York, N. Y. and on March 4, 1953 at Detroit, Mich. The committee now consists of 110 members, of whom 49 are classified as producers, 35 as consumers and 26 as general interest members.

The committee voted by letter ballot on elimination of the Uniformity of Coating (Preece Test) requirements from A 122 - 52 T and A 218 - 52 T. The results of this letter ballot (51 affirmative, 10 negative) indicated the need for further study of this matter. The appended report of tests made at the National Bureau of Standards on the Preece Test are a first step in this direction.[1]

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Zinc-Coated Steel Overhead Ground Wire Strand (A 363 - 52 T).

Revision and Reversion to Tentative of Standard Specifications for:

Zinc-Coated Steel Wire Strand ("Galvanized" and Class A ("Extra Galvanized")) (A 122 - 41), and

Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (A 218 - 41).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the tentative specifications appear in the 1952 Book of ASTM Standards, Part 1.

The new tentative A 363 - 52 T specifies a strand without splices. The revisions of A 122 and A 218 eliminate the minimum length of lay requirements to conform with manufacturing practices of all producers when making preformed strand.

REVISION OF TENTATIVES

The committee recommends that the following two tentative specifications be revised as indicated and continued as tentative:

Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (**A 93 - 52 T**).[2]—The inclusion of coils in these specifications necessitated some clarifying revision of the paragraphs on Finish and Inspection. In addition, tables were expanded to include all gage numbers and provision made for sampling of coils narrower than the 2.25 in. test samples. The following changes are recommended:

Section 1.—Under Class "D" omit the second sentence, which reads as follows: "These coatings approximate those of Class C except in medium gages in which coatings of Class D are appreciably lighter."

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28-July 3, 1953.

[1] See p. 125.

[2] 1952 Book of ASTM Standards, Part 1.

Table II.—Amplify to include gages Nos. 15, 17, 19, 21, 23, and 25, making the table read as follows:

TABLE II.—ORDERED WEIGHT OF COATINGS, OUNCES PER SQUARE FOOT.

Galvanized Sheet Gage Number	Class A	Class B	Class C	Class D	Class E
8	2.75	2.50	2.00	No Specified Coatings, See Section 1.	No Specified Coatings, See Section 1.
9	2.75	2.50	2.00		
10	2.75	2.50	2.00		
11	2.75	2.50	2.00		
12	2.75	2.50	2.00		
13	2.75	2.50	2.00		
14	2.75	2.50	2.00		
15	2.75	2.50	2.00		
16	2.75	2.50	2.00		
17	2.75	2.50	1.75		
18	2.75	2.50	1.75		
19	2.75	2.50	1.75		
20	2.75	2.50	1.75		
21	2.75	2.50	1.75		
22	2.75	2.50	1.75		
23	..	2.50	1.50		
24	..	2.50	1.50		
25	..	2.25	1.25		
26	..	2.25	1.25		
27	..	2.00	1.25		
28	..	2.00	1.25		
29	..	1.50	1.25		
30	..	1.25	1.25		

Table III.—Amplify to include intermediate gages. The additions are as follows:

TABLE III.—COATING BEND TESTS.

Ordered Coating (Pot Yield), oz per sq ft	Number of Pieces of Same Gage Used in Coating Bend Tests							
	Galvanized Sheet Gage							
	No. 17	No. 18	No. 19	No. 21	No. 23	No. 25	No. 27	No. 29
2.00	..	..	..	..	8	9	9	..
1.75	7	7	7	8	8	9	9	..
1.50	6	6	6	6	6	6	6	6
1.25 (Including Class D)	5	5	5	6	6	6	6	6

Table IV.—Add column of Mean Thickness, in., as follows:

0.1681
0.1532

0.1382
0.1233
0.1084
0.0934
0.0785

0.0710
0.0635
0.0575
0.0516
0.0456

0.0396
0.0366
0.0336
0.0306
0.0276

0.0247
0.0217
0.0202
0.0187
0.0172

0.0157
0.0142
0.0134

Section 10(b).—Add the following sentence: "For coils narrower than 2.25 in., test samples shall be of such a length as to render the area of the sample equal to 5.06 sq in."

Section 17.—Revise to read as follows:

(*a*) The sheets shall be of prime finish, that is, free from injurious defects, such as blisters, flux, and uncoated spots.

(*b*) Coils commonly contain some abnormal defects such as welds to join coils, holes, etc., which render a portion of the coil unusable since the inspection of coils does not afford the same opportunity to reject portions containing defects as is the case with cut lengths.

Section 18(a).—Revise to read as follows:

"(*a*) *Cut Length Sheets.*—The top sheet of each bundle or the top sheet of a number of sheets when shipped loose, shall, unless otherwise ordered, show legibly the name and brand of the manufacturer, gage, size, and ordered coating, except for Classes "D" and "E."

Section 19.—Revise to read as follows:

"The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works that concern the manufacture of the material ordered. The manufacturer shall afford the inspector all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification. All inspection and tests (except check analysis) shall be made prior to shipment at the manufacturer's works, unless otherwise specified, and such inspection or sampling shall be made in conjunction with, and to the extent of, the manufacturer's regular inspection operations."

Section 20.—Revise to read as follows:

"If the purchaser elects to test and inspect the material at its destination, sheets and coils represented by specimens that fail to conform to the requirements of these specifications will be rejected, and the manufacturer will be notified within two weeks of receipt of shipment by the purchaser."

Tentative Specifications for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (**A 308 – 50 T**).[2]—The following changes are recommended:

Section 9(b).—Add the following sentence: "For coils narrower than 2.25 in., test samples shall be of such a length as to render the area of the sample equal to 5.06 sq in."

Section 16.—Revise to read as follows:

> (*a*) The sheets shall be free from injurious defects such as laminations, blisters, slivers, pits from rolled-in scale, uncoated or imperfectly coated spots readily observable by the unaided eye, deep scratches or heavy list edges.
>
> (*b*) Coils commonly contain some abnormal defects such as welds to join coils, holes, etc., which render a portion of the coil unusable since the inspection of coils does not afford the same opportunity to reject portions containing defects as is the case with cut lengths.

Section 17.—Revise to read as follows:

> "(*a*) *Cut Lengths Sheets*.—The top sheet of each bundle or the top sheet of a number of sheets when shipped loose, shall, unless otherwise ordered, show legibly the name and brand of the manufacturer, gage, size, and ordered coating weight, if specified.
>
> "(*b*) *Coils*.—Coils or bundles of coils shall be individually tagged showing ordered coating weight, if specified, thickness, width, and weight of coil."

Section 18.—Revise to read as follows:

> "The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works that concern the manufacture of the material ordered. The manufacturer shall afford the inspector all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification. All inspection and tests (except check analysis) shall be made prior to shipment at the manufacturer's works unless otherwise specified, and such inspection or sampling shall be made in conjunction with, and to the extent of, the manufacturer's regular inspection operations."

Section 19.—Revise to read as follows:

> "If the purchaser elects to test and inspect the material at its destination, sheets and coils represented by specimens that fail to conform to the requirements of these specifications will be rejected, and the manufacturer will be notified within two weeks of receipt of shipment by the purchaser."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends the immediate revision in three standards as set forth below and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Methods of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (**A 90 – 39**).[2]—This standard was revised to eliminate references to methods of sampling, provide for the use of samples other than 2.25 in. square, add the dilute hydrochloric acid method for determining weight of coating, and to add safety warnings. The revised methods are appended hereto.[3]

Standard Specifications for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars, and Their Products **(A 123 – 47).**[2]—The following changes are recommended:

Title.—Change to read "Standard Specifications for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip.

Section 1(a).—Revise to read as follows:

> (*a*) These specifications cover the protective zinc coatings, applied on products fabricated from rolled, pressed and forged steel shapes, plates, bars, and strip, $\frac{1}{8}$ in. thick and heavier (Note 1) by dipping the articles in a molten bath of zinc (Note 2).

Section 6.—Renumber present Section 6 as 6(*a*) and add the following new Paragraph (*b*):

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[3] The revised standard was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 1.

(*b*) The galvanizing of bolts and similar threaded fasteners shall be in accordance with Standard Specifications for Zinc Coating (Hot Dip) on Iron and Steel Hardware (ASTM Designation: A 153).

NOTE.—One ounce of zinc per sq ft of surface, based upon mathematical calculation, corresponds to a coating thickness of 0.0017 in.

Section 10.—Revise to read as follows:

After immersion in the molten zinc, the shapes, plates, bars, or strip shall not be subjected to any process of scraping or wiping which will reduce the uniformity or the specified weight of the zinc coating.

Note 5.—Delete.

Standard Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (**A 153 – 49**).[2]—The following changes are recommended:

Section 1.—Revise to read as follows:

These specifications cover zinc coatings applied by the hot-dip process on iron and steel hardware and light shapes (such as used for steel windows).

Section 5.—Substitute the word "material" for the word "hardware" in the last sentence.

Section 6.—For Class A Material change the Minimum Weight of Coating in oz per sq ft of surface from "1.80" to "2.00" for average of specimens tested and from "1.60" to "1.80" for any individual specimen.

For Class C Material add the following at the end of the information in the first column of the table: "Washers $\frac{3}{16}$ in. and $\frac{1}{4}$ in. thick."

For Class D Material add the following at the end of the information in the first column of the table: "Washers under $\frac{3}{16}$ in. thick."

Note.—Renumber as "Note 1" and add the following Notes 2 and 3:

NOTE 2.—One ounce of zinc per sq ft of surface, based upon mathematical calculation, corresponds to a coating thickness of 0.0017 in.

NOTE 3.—The weight and uniformity of zinc coating for light steel shapes. such as used for steel windows, shall conform to Class B-2 in the above table if the thickness is under $\frac{3}{16}$ in. If the thickness is $\frac{3}{16}$ in. and over it shall conform to Class B-1 of the table.

Section 8.—Renumber as Section 8(*a*). Delete the last sentence. Add the following new Paragraphs (*b*), (*c*), and (*d*):

(*b*) A convenient bend test for embrittlement of galvanized hardware such as bolts, pole and tower steps, insulator clevises, braces, rods, etc. consists of clamping one end of the article in a vise and bending the other end 90 degrees. A suitable length of pipe, used as a lever, is very helpful. An embrittled article will not withstand a 90-deg bend. On the other hand, an article in the manufacture of which proper precautions against embrittlement have been taken will withstand such bending. If such a test is made on threaded articles it shall be made on the unthreaded portion.

(*c*) When the article is of such shape or size as not to accommodate bending, it may be struck a sharp blow with a 2-lb hammer. If the steel is not embrittled it should not crack under such a blow.

(*d*) Material found to be embrittled by either of the above tests shall be rejected.

Appendix.—Add the following Appendix:

A corrosion-resistant high-strength low-alloy steel (ASTM Designation: A 242), if so specified or approved by the purchaser, may be used. The suitability of the chemical composition to resist atmospheric corrosion shall be based upon evidence acceptable to the purchaser.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation in present form of Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 51 T),[3a] subject to approval of Committee B-8.

The committee recommends the continuation in present form of Tentative Specification for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized Before Weaving (A 337 – 51 T).[2]

Both of these specifications will be reviewed for adoption as standard this next year.

[3a] 1952 Book of ASTM Standards Part 2.

WITHDRAWAL OF TENTATIVE

The committee recommends dropping Specification for Lead-Alloy Coating on Iron or Steel Hardware (A 267 – 46 T).[2] This specification, a war measure, was continued as information, but is not now being used. Although this specification is to be discontinued, it should still be listed in the Index to Standards.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Test (O. B. Ellis, chairman) completed its work on the revision of Standard Methods of Test for Weight of Coating on Zinc-Coated Iron or Steel Articles (A 90 – 39) and submitted it to Committee A-5 for letter ballot. The Standard Method of Test for Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test (A 309 – 49) is being studied with the view to including other methods of test. The subcommittee has under study proposals to prepare a specification for the determination and analysis of tin coatings on tin plates, as suggested by the American Iron and Steel Institute. The subcommittee is also investigating non-destructive methods for the determination of thickness of metallic coatings and the accuracy of test methods using magnetic gages. Work initiated several years ago on the Preece Test has been completed by the National Bureau of Standards and is presented in an appendix to this committee report.[1]

Subcommittee XI on Sheet Specifications (E. F. Lundeen, chairman) conducted letter ballots on revision of Tentative Specifications for Zinc-Coated Iron or Steel Sheets, Coils and Cut Lengths (A 93 – 52 T) and Tentative Specifications for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 – 50 T). The inclusion of coils in these specifications necessitated revisions of sections on Finish and Inspection. Those matters not controversial were later resolved in committee with editorial changes and submitted to A-5 for ballot. The controversial section having to do with number and degree of defects in coils will be studied further.

Subcommittee XII on Wire Specification (J. F. Occasione, chairman). The proposed revision of Standard Specification for Zinc-Coated (Galvanized) Iron or Steel Tie Wires (A 112 – 33) has progressed to the point where clarification of the sampling procedure is all that is required to permit issuance of a draft specification. The proposed revision of Specification for Zinc-Coated Iron or Steel Chain Link Fence Fabric Galvanized After Weaving (A 117 – 33) has been delayed by lack of consensus on size tolerance, and further discussion indicated a desire to review the Uniformity of Coating (Preece) Test requirement with the thought of deleting same. The proposal to delete the Uniformity of Coating (Preece) Test requirements from Specifications A 122 and A 218 was remanded to Subcommittee because of the results of letter ballot in Committee A-5. This proposal will be reviewed at a future time after the results of a forthcoming Bureau of Standards report on a re-investigation of the Preece tests are available for consideration. The subcommittee voted to retain Specification A 337 – 51 T as a tentative specification. The Section on Strand will determine whether the new Specification for Zinc-Coated Overhead Ground Wire Strand (A 363 – 52 T) should be modified to include additional constructions or sizes. This section will also make recommendations relative to changes in Specifications A 122 and A 218 made necessary by the issuance of A 363 – 52 T. Subcommittee XII will initiate work on the preparation of specifications for zinc-coated armor wire and for zinc-coated poultry and turkey run netting.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) recommends that Specification for Lead-Alloy Coating on Iron and Steel Hardware (A 267 – 46 T) be dropped because lead coating on hardware is not now being used commercially. Specifications for Zinc Coatings on Structural Steel Shapes, Plates and Bars and Their Products (A 123 – 47) and Zinc Coating on Iron and Steel Hardware (A 153 – 49) have been revised and submitted to A-5 for letter ballot. The subcommittee is studying a Recommended Practice for Safeguarding Against Warpage and Distortion During Hot Dip Galvanizing of Steel Assemblies, also a specification for zinc coating (hot dip) on miscellaneous fabricated or assembled steel products.

Subcommittee XIV on Sheet Tests (C. P. Larrabee, chairman) makes detailed reports of its inspection activities in the even numbered years. The atmospheric tests of corrugated sheets at Annapolis, Md., Altoona, Pa., and State College, Pa., were inspected in April, 1952, and again in April, 1953.

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) has continued with the field inspections. A detailed report of the results of 1951 and 1952 inspection is appended to this report.[5] The test site at Sandy Hook, N. J., has been abandoned due to extensive damage to the specimens.

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman). The entire membership of Committee A-5 and a subcommittee of the Edison Electric Institute have been canvassed relative to a proposed new hardware test. Over 100 questionnaires were sent out and 29 replies in favor of a new test were received. It is concluded that a new test is warranted.

This report has been submitted to letter ballot of the Committee, which consists of 139 members; 96 members returned their ballots, of whom 82 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. P. JAHN,
Chairman.

C. P. LARRABEE,
Secretary.

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[5] See p. 102.

REPORT OF SUBCOMMITTEE XV ON ATMOSPHERIC EXPOSURE TESTS OF WIRE AND WIRE PRODUCTS

(COVERS 1951 AND 1952 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1952 and presents in detail the data from 1951 and 1952 inspections. Reference to earlier reports of Committee A-5 on Corrosion of Iron and Steel which appeared in the Society's *Proceedings* for 1939, 1941, 1943, 1945, 1947, 1949, and 1951 will be helpful in studies of these data. These earlier reports contain descriptions of the test methods and micrographs and other characterizations of specimens in the test as well as the data assembled during previous inspections.

The materials under test are mostly bare steel and zinc-coated wires and wire products but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For ready comparisons of coating thickness 1 oz per sq ft of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0013 in. thick for copper coatings, and 0.0010 in. thick for lead coatings.[1]

Specimens of wires and wire products have been exposed for about 16 yr at eleven locations. The location of these sites and their general classification regarding atmospheres are as follows:

Test Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa.	Severe industrial	On Brunot Island in the Ohio River about 2 miles west of the city.
Bridgeport, Conn.	Industrial	In the city.
Sandy Hook, N. J.	Seacoast	About 300 yd from Atlantic Ocean.
Santa Cruz, Calif.	Seacoast	About 3 miles from Pacific Ocean.
State College, Pa.	Rural	Central Pennsylvania.
Lafayette, Ind.	Rural	Wabash River Valley.
Ames, Iowa	Rural	Central Iowa.
Manhattan, Kans.	Rural	Kansas River Valley.
Ithaca, N. Y.	Rural	Central New York.
College Station, Tex.	Rural	About 120 miles inland.
Davis, Calif.	Rural	About 80 miles inland.

The Pittsburgh and Sandy Hook sites were abandoned in 1951 and 1952, respectively.

At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations; and chain link fence at eight locations.

The extent of corrosion is being measured by weight loss determinations, by visual examination of all specimens at the test site, and by tension tests on wire specimens. The Pittsburgh, Bridgeport, Sandy Hook, and State College specimens were inspected by a traveling committee. Inspections were made at the other test sites by the university people in charge of the test plots.

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analysis of lead coatings.

TABLE I. - REPORT OF 1951 AND 1952 INSPECTIO

Coating Group	Average Coating, oz. per sq.ft.of Surface[a]	Wire	State College, Pa.		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Cal.		College Sta., Tex.		Davis, Cal.	
			14.30 Yr.	15.30 Yr.	15.00 Yr.	16.00 Yr.	14.88 Yr.	15.82 Yr.	14.93 Yr.	15.88 Yr.	14.91 Yr.	15.90 Yr.	14.83 Yr.	15.80 Yr.	14.83 Yr.	15.80 Yr.	14.85 Yr.	15.81 Yr.
0.20 to 0.30 Avg. 0.28	0.27	No. 512	100R	100R	100R	100R	100R	100R	70R	20R	100R	100R	100R	100R	100R	100R	60R	64R
	0.30	No. 410	100R	100R	100R	100R	100R	100R	100R	50R	100R	100R	63R	100R	100R	100R	70R	92R
	0.28	No. 411	100R	100R	100R	100R	100R	100R	30R	20R	100R	100R	25R	44R	95R	100R	3R	3R
	0.29	No. 510	100R	100R	100R	100R	100R	100R	50R	15R	100R	100R	47R	89R	100R	100R	5R	6R
	0.29	No. 412	100R	100R	100R	100R	100R	100R	10R	25R	100R	100R	32R	39R	100R	100R	3R	3R
	0.24	No. 511	100R	100R	100R	100R	100R	100R	50R	30R	100R	100R	64R	96R	100R	100R	30R	44R
0.25 to 0.35 Avg. 0.30	0.25	No. 315	100R	100R	100R	100R	100R	100R	50R	45R	100R	100R	93R	94R	100R	100R	65R	94R
	0.28	No. 215	100R	100R	100R	100R	100R	100R	60R	40R	100R	100R	71R	69R	100R	100R	65R	87R
	0.27	No. 117	100R	100R	100R	100R	100R	100R	30R	40R	100R	100R	28R	46R	100R	100R	20R	26R
	0.38	No. 317	100R	100R	100R	100R	109R	100R	GY	G	100R	100R	4R	GY	13R	12R	G	G
	0.36	No. 217	100R	100R	100R	100R	100R	100R	25R	30R	100R	100R	18R	17R	53R	76R	1R	1R
	0.32	No. 316	100R	100R	100R	100R	100R	100R	5R	10R	100R	100R	19R	20R	38R	19R	2R	2R
	0.30	No. 216	100R	100R	100R	100R	100R	100R	5R	5R	100R	100R	20R	25R	33R	48R	1R	GY
	0.25	No. 115	100R	100R	100R	100R	100R	100R	60R	50R	100R	100R	68R	89R	100R	100R	30R	42R
0.35 to 0.45 Avg. 0.38	0.36	No. 520	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	23R	25R	100R	100R	1R	1R
	0.35	No. 422	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	8R	1R	12R	13R	G	G
	0.31	No. 423	100R	100R	100R	100R	100R	100R	5R	1R	100R	100R	11R	GY	14R	14R	G	G
	0.44	No. 522	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	9R	GY	11R	13R	G	G
	0.37	No. 282	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	7R	GY	14R	8R	G	G
	0.38	No. 521	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	14R	GY	20R	21R	G	G
	0.47	No. 421	100R	100R	100R	100R	100R	100R	10R	10R	100R	100R	19R	1R	22R	24R	G	G
0.45 to 0.55 Avg. 0.47	0.44	No. 325	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	10R	GY	GY	GY	G	G
	0.50	No. 228	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	8R	GY	GY	GY	G	G
	0.54	No. 329	100R	100R	100R	100R	60R	89R	GY	GY	100R	100R	11R	GY	14R	14R	G	G
	0.52	No. 227	100R	100R	100R	100R	65R	97R	GY	GY	100R	100R	10R	GY	GY	GY	G	G
	0.41	No. 125	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	14R	GY	24R	15R	1R	1R
	0.54	No. 127	100R	100R	100R	100R	75R	98R	GY	GY	100R	100R	10R	GY	16R	5R	G	G
	0.38	No. 326	100R	100R	100R	100R	90R	100R	GY	GY	100R	100R	3R	GY	GY	GY*	G	G
	0.45	No. 226	100R	100R	100R	100R	25R	83R	3R	GY	100R	100R	6R	GY	GY	GY	G	G
	0.47	No. 327	100R	100R	100R	100R	95R	100R	GY	GY	100R	100R	7R	GY	GY	GY	G	G
	0.45	No. 225	100R	100R	100R	100R	50R	94R	GY	GY	100R	100R	6R	GY	GY	GY	G	G
0.50 to 0.60 Avg. 0.56	0.61	No. 530	100R	100R	100R	100R	100R	100R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
	0.57	No. 432	100R	100R	100R	100R	50R	100R	MG	MG	100R	100R	MG	MG	G	GY*	MG	MG
	0.60	No. 531	100R	100R	100R	100R	100R	100R	G	G	100R	100R	MG	MG	G	G	G	G
	0.48	No. 431	100R	100R	100R	100R	100R	100R	G	GY	100R	100R	10R	GY	Y	GY	G	G
	0.54	No. 430	100R	100R	100R	100R	15R	49R	G	G	100R	100R	9R	GY	Y	5R	G	G
0.60 to 0.70 Avg. 0.65	0.69	No. 338	50R	90R	100R	100R	G	G	MG	MG	100R	100R	MG	MG	G	G	MG	MG
	0.68	No. 236	50R	95R	100R	100R	G	2R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
	0.61	No. 335	100R	100R	100R	100R	95R	99R	MG	G	100R	100R	MG	G	G	GY*		G
	0.63	No. 237	95R	100R	100R	100R	15R	37R	MG	MG	95R	100R	MG	MG	G	G	MG	MG
	0.74	No. 135	99R	100R	100R	100R	G	3R	MG	G	100R	100R	MG	G	G	GY*	G	MG
	0.57	No. 337	100R	100R	100R	100R	25R	63R	GY	GY	100R	100R	GY	GY	GY	GY*	1R	G
	0.67	No. 235	100R	100R	100R	100R	G	G	GY	GY	100R	100R	GY	GY	GY	GY*	G	G
	0.65	No. 239	100R	100R	100R	100R	3R	5R	GY	GY	100R	100R	GY	GY	GY	GY	G	G
	0.67	No. 138	100R	100R	100R	100R	5R	6R	GY	GY	100R	100R	GY	GY	2R	5R	G	G
	0.64	No. 139	100R	100R	100R	100R	15R	24R	GY	GY	100R	100R	GY	GY	10R	GY	G	G
	0.66	No. 336	100R	100R	100R	100R	10R	24R	GY	5R	100R	100R	GY	GY	Y	GY*	G	G
	0.60	No. 238	100R	100R	100R	100R	15R	54R	GY	GY	100R	100R	GY	GY	GY	GY	G	G
0.70 to 0.85 Avg. 0.75	0.70	No. 540	45R	85R	100R	100R	5R	36R	MG	MG	99R	100R	MG	MG	G	G	MG	MG
	0.72	No. 442	60R	100R	100R	100R	G	2R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
	0.79	No. 543	80R	95R	98R	100R	20R	45R	MG	MG	95R	99R	G	G	G	G	G	G
	0.78	No. 441	100R	100R	100R	100R	10R	12R	MG	MG	100R	100R	G	G	G	G	G	G
	0.70	No. 542	100R	100R	100R	100R	80R	100R	MG	G	100R	100R	MG	MG	G	G	G	G
	0.74	No. 443	90R	95R	100R	100R	10R	24R	MG	MG	99R	100R	MG	MG	G	G	G	G
	0.74	No. 495	20R	60R	100R	100R	G	1R	G	G	99R	100R	-	-	Cr	Cr	-	-
	0.76	No. 541	100R	100R	100R	100R	G	1R	G	G	100R	100R	GY	GY	Y	GY*	G	G
	0.76	No. 440	100R	100R	100R	100R	1R	9R	G	GY	100R	100R	GY	GY	Y	GY*	G	G
	0.87	No. 444	100R	100R	100R	100R	G	1R	GY	GY	100R	100R	GY	GY	Y	GY*	G	G
0.80 to 1.00 Avg. 0.88	1.05	No. 348	G	G	G	G	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG
	0.81	No. 248	55R	100R	100R	100R	G	2R	MG	MG	100R	100R	MG	MG	G	G	MG	MG
	0.88	No. 347	25R	90R	90R	100R	1R	6R	MG	MG	15R	80R	G	G	G	G	G	G
	0.95	No. 246	1R	10R	55R	80R	G	2R	MG	MG	15R	63R	G	G	G	G	MG	MG
	0.88	No. 346	100R	100R	100R	100R	G	1R	MG	G	99R	100R	G	G	G	G	G	G
	0.81	No. 245	90R	98R	99R	100R	G	G	MG	G	100R	100R	G	MG	G	GY*	G	G
	0.81	No. 296	GY	15R	95R	100R	G	G	MG	G	70R	99R	-	-	-	-	-	-
	0.90	No. 148	50R	95R	95R	100R	G	2R	MG	MG	90R	99R	MG	MG	G	G	G	G
	0.85	No. 149	35R	80R	95R	100R	G	28R	MG	MG	80R	99R	G	G	G	G	MG	MG
	0.84	No. 345	100R	100R	100R	100R	G	G	GY	GY	98R	100R	GY	GY	GY	GY*	G	G
	0.96	No. 247	60R	90R	100R	100R	G	G	GY	GY	35R	85R	GY	GY b	GY	GY b	G	G b
Avg. 1.28	1.35	No. 800	GY	Y	Y	90R	G	5R	MG	MG	GY	Y	Sandy Hook, N.J.[c]		Bridgeport, Conn.[c]			
	1.20	No. 801	GY	Y	10R	65R	G	1R	MG	MG	GY	99R	14.44 Yr.*			14.45 Yr.	15.45 Yr.	
1.60 to 1.80 Avg. 1.76	2.08	No. 552	G	G	G	G	G	G	MG	MG	G	G	G			15R	98R	
	1.83	No. 451	G	G	G	G	G	G	MG	MG	G	G	G			100R	100R	
	1.75	No. 350	G	G	G	G	G	G	MG	MG	G	G	G			GY	70R	
	1.69	No. 252	G	G	G	G	G	G	MG	MG	G	G	G			35R	100R	
	1.65	No. 351	G	G	1R	2R	G	G	MG	MG	G	1R	10R			65R	99R	
	1.76	No. 250	G	G	G	G	G	G	MG	MG	G	GY	15R			30R	75R	
	1.69	No. 551	G	G	G	G	G	G	MG	MG	G	G	G			50R	85R	
	1.50	No. 450	G	G	GY	G	G	G	MG	MG	G	GY	35R			100R	100R	
	1.75	No. 352	G	G	GY	4R	G	G	MG	MG	G	G	20R			15R	70R	
	1.86	No. 251	G	GY	GY	G	G	G	MG	MG	G	1R	35R			45R	60R	
	1.86	No. 151	1R	2R	3R	10R	G	G	MG	MG	G	9R	45R			40R	50R	
heavy Avg. 2.89	2.80	No. 397	G	G	G	G	G	G	MG	MG	G	G	G			GY	GY	
	2.72	No. 298	G	G	G	G	G	G	MG	MG	G	G	G			GY	GY	
	3.00	No. 299	G	G	G	G	G	G	MG	MG	G	G	G			GY	GY	

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

b All other coating weight groups G or MG.

[c] All other coating weight groups were 100R.

* Specimens destroyed shortly after this inspection.

IONS OF UNFABRICATED WIRE SPECIMENS.

PLATE I
PROCEEDINGS, AM. SOC. TESTING MATS.
VOL. 53
REPORT OF COMMITTEE A-5:
WIRE INSPECTION REPORT

al.
5.81 Yr.
84R
92R
3R
6R
3R
44R
94R
87R
26R
G
1R
2R
GY
42R
1R
G
G
G
G
G
G
G
G
G
1R
G
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G
G
MG
MG
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MG
G
G
-
G
MG
G
G b
r.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. Cr = yellow from chromate treatment. MG = intermediate between M and G. Gy = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.
* Speckled appearance.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic, G = gray, Y = yellowed, R = rust of base metal, PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance. D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY predominately gray but showing indication of Y. SY* or SR* indicate, respectively: superficial Y and superficial, R, the asterisk denoting a speckled appearance. Figures (as 5, 20, 50, 100) are percentages. R+Y = indistinguishable proportions of R and Y.

	Gage	Wire	* Sandy Hook, N J.	Bridgeport, Conn.		State College, Pa.		College Sta., Texas		Davis, Calif,	
			14.44 Yr.	14.45 Yr.	15.30 Yr.	14.30 Yr.	15.30 Yr.	14.83 Yr.	15.79 Yr.	14.84 Yr.	15.87 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	Gn	Gn	Gn	Gn-B	Gn	Br	Br	Br	Br
	9	No. 022, 9.24-oz.	Gn	Gn	Gn	Gn-B	Gn	Br	Br	Br	Br
Lead-coated Wires	6	No. 040, 1.40-oz.	100R+Y	GYPHR	GY	100R+Y	100R+Y	20R+Y	21R+Y	20SR	19SR
	9	No. 041, 1.46-oz.	100R+Y	GYPHR	GY	100R+Y	100R+Y	55R+Y	56R+Y	70SR	60SR
	11	No. 042, 1.68-oz.	100R+Y	GYPHR	GY	100R+Y	100R+Y	35R+Y	35R+Y	25SR	23SR
	14-1/2	No. 043, 1.90-oz.	100R+Y	GYPHR	GY	100R+Y	100R+Y	5R+Y	6R+Y	20SR	18SR
Corrosion-Resistant Uncoated Wires:											
12-14% Cr., Cold drawn	12	No. 001	YSR	GY	Y	MG	MG	100R+Y	3R+Y	40SR	41SR
	9	No. 004	YSR	GY	Y	MG	MG	96R+Y	1R+Y	40SR	26SR
	17	No. 005	YSR	GY	Y	MG	MG	100R+Y	27R+Y	50SR	46SR
12-14% Cr., air quenched	13	No. 002	YSR	GY	GY	MG	MG	88R+Y	6R+Y	19SR	19SR
	10	No. 006	YSR	GY	GY	MG	MG	78R+Y	56R+Y	16SR	16SR
	18	No. 007	YSR	GY	GY	MG	MG	92R+Y	4R+Y	40SR	40SR
18% Cr., 8% Ni.	15	No. 003	MG	MG	MG	M	M	MGTrY	MGTrY	MG	MG
	12	No. 008	MG	MG	MG	M	M	MGTrY	MGTrY	MG	MG
	20	No. 009	MG	MG	MG	M	M	MGTrY	MGTrY	MG	MG
	14	No. 015	MG	MG	MG	M	M	MGTrY	MGTrY	-	-

	Gage	Wire	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Calif.	
			15.00 Yr.	16.00 Yr.	14.68 Yr.	15.62 Yr.	14.93 Yr.	15.98 Yr.	14.91 Yr.	15.90 Yr.	14.83 Yr.	15.80 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	Gn	Gn	Br	Br	Br	Br	B	Gn	Br	Br
	9	No. 022, 9.24-oz.	Gn	Gn	Br	Br	Br	Br	B	BrGn	Br	Br
Lead-coated Wires	6	No. 040, 1.40-oz.	75R+Y	50PHR	17R	9R	10R*	5PHY	40R+Y	42R+Y	20SR	18SR
	9	No. 041, 1.46-oz.	60R+Y	40PHR	44R	50R	25R	20R	40R+Y	40R+Y	60SR	61SR
	11	No. 042, 1.68-oz.	75R+Y	50PHR	32R	64R	10R	15R	30R+Y	30R+Y	20SR	23SR
	14-1/2	No. 043, 1.90-oz.	60R+Y	40PHR	39R	69R	G	GY	20R+Y	20R+Y	30SR	36SR
Corrosion-Resistant Uncoated Wires:												
12-14% Cr., Cold drawn	12	No. 001	M	M	SY	1RSY	MG	M	Y*	SY*	90SR	87SR
	9	No. 004	M	M	SY	2RSY	MG*	M	Y*	SY*	70SR	69SR
	17	No. 005	M	M	SY	SY	MG*	M	Y*	SY*	80SR	88SR
12-14% Cr., air quenched	13	No. 002	M	M	SY	SY	MG	M	M*	SY*	30SR	26SR
	10	No. 006	M	M	SY	SY	MG	M	M*	SY*	25SR	26SR
	18	No. 007	M	M	SY	SY	MG	M	Y*	SY*	70SR	67SR
18% Cr., 8% Ni.	15	No. 003	M	M	SY	SY	M	M	M	M	MG	MG
	12	No. 008	M	M	SY	SY	M	M	M	M	MG	MG
	20	No. 009	M	M	SY	SY	M	M	M	M	MG	MG
	14	No. 015	M	M	SY	SY	M	M	M	M	-	-

* Specimens destroyed shortly after this inspection

TABLE A.—REPORT OF GROUP AVERAGES THROUGH 1952—16-YR EXPOSURE.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y. Figures preceding R (as 5, 20, 50, 100) are percentages. Italized readings are for those groups which as a group have not reached 1R or 100R.

Coating Type or Weight Zn	No. in Group	Pittsburgh (14 yr)		Sandy Hook (15 yr)		Bridgeport		State College		Lafayette		Ithaca		Ames		College Station		Manhattan		Santa Cruz		Davis	
		Average of Observed Years of Exposure until 1R and to 100R—or State of Corrosion after Approximately 16-yr Exposure																					
		1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R	1R	100R
*Unfabricated Wires																							
0.20 to 0.30	6	0.8	1.3	1.7	2.8	2.1	2.8	5.1	6.2	4.6	5.6	5.6	6.2	6.3	7.5	8.5	12.8	10.4	*27R*	12.6	*78R*	12.3	*40R*
0.25 to 0.35	8	0.9	1.4	2.5	3.2	2.3	3.5	4.9	6.7	4.8	6.3	5.6	7.3	6.9	9.3	9.0	*70R*	10.3	*40R*	12.3	*45R*		*35R*
0.35 to 0.45	7	1.5	2.0	2.8	3.7	2.9	3.7	6.6	8.4	5.5	6.8	6.7	8.3	8.4	11.4	11.5	*28R*		*2R*		*4R*		*G*
0.45 to 0.55	10	1.8	2.5	3.8	5.2	3.8	5.6	7.9	10.3	7.1	9.2	8.3	10.4	10.7	*96R*		*3R*		*GY*		*GY*		*G*
0.50 to 0.60	5	1.7	2.1	4.7	6.0	4.7	5.7	9.5	11.7	8.5	10.0	9.3	11.2	12.1	*90R*		*1R*		*G*		*G*		*G*
0.60 to 0.70	12	2.2	2.8	5.6	6.8	5.4	7.1	10.2	13.2	8.8	11.4	10.4	13.0		*27R*		*GY*		*GY*		*GY*		*G*
0.70 to 0.85	10	2.1	2.8	5.9	7.2	5.7	7.1	11.3	*90R*	9.8	11.9	11.1	13.3		*23R*		*G*		*G*		*G*		*G*
0.80 to 1.00	11	2.7	3.3	7.1	8.7	6.8	8.9		*80R*		*90R*		*83R*		*4R*		*G*		*G*		*G*		*G*
1.20 to 1.35	2	3.7	5.0	9.4	11.5	9.0	11.5		*Y*		*78R*		*Y*		*3R*		*G*		*MG*		..		..
1.60 to 1.80	11	4.2	5.6		*10R*		*80R*		*G*		*1R*		*G*		*G*		*G*		*MG*		*MG*		*MG*
2.70 to 3.00	3	7.3	8.4		*G*		*GY*		*G*		*G*		*G*		*G*		*G*		*MG*		*MG*		*MG*
Cu Coat	2		*D*		*Gn*		*Gn*		*Gn*		*Gn*		*Gn*		*Br*		*Br*		*Br*		*Br*		*Br*
Pb Coat	4		*D*		*100R +Y*		*GY*		*100R +Y*		*50PHR*		*40R*		*50R*		*30R*		*10R*		*30R*		*30SR*
12–14% Cr	6		*D*		*YSR*		*Y*		*MG*		*M*		*SY*		*SY*		*10R +Y*		*MG*		*60R*		*30SR*
18% Cr–8% Ni	3		*D*		*MG*		*MG*		*M*		*M*		*M*		*SY*		*MG*		*M*		*MG*		*MG*
Wire Strand																							
0.69 to 0.93	4	4.0	6.8	9.4	*60R*	9.9	15.3		*GY*	15.0	*15R*		*3R*		*G*		*G*		*MG*		*G*		*G*
1.03 to 1.17	3	4.9	7.0	11.9	*40R*		*30R*		*GY*		*15R*		*GY*		*G*		*G*		*MG*		*MG*		*MG*
1.32 to 1.51	4	6.0	10.0	14.1	*10R*		*10R*		*GY*		*20R*		*1R*		*G*		*G*		*MG*		*MG*		*MG*
1.71 to 2.47	1	8.5	*40R*^{e}		*G*		*G*		*G*		*G*		*G*		*G*		*G*		*MG*		*MG*		*MG*
Pb Coat	1	5.0^d	*D*		*100R+Y*		*20R*		*10R*		*50PHR*		*70R*		*60R*		*10PHR*		*25R*		*60SR*		*10R*
Chain Link Fence																							
0.29 to 0.52^a	3	1.5	2.2	2.2	4.5	2.9	4.8	6.4	9.1	5.7	9.0	5.9	11.5							12.7	*20R*	12.8	*20R*
1.13 to 1.22^a	2	3.7	4.7	8.4	10.6	8.9	12.0	14.5	*2R*	14.0	*50R*		*GY*		No Specimens Exposed		No Specimens Exposed		No Specimens Exposed		*MG*		*MG*
2.09 to 2.92^a	2	6.5	7.8		*G*		*G*		*G*		*G*		*MG*								*MG*		*MG*
1.40 to 1.97^b	10	3.7	6.4	9.0	12.3	9.9	13.0		*GY*	15.5	*20R*		*10R*								*G*		*MG*
4.21^b	1	7.6	*50R*^{e}		*GY*		*GY*		*G*		*GY*		*GY*								*MG*		*MG*
Cu Coat	1		*Gn*^{c}		*Gn*		*Gn*		*Gn*		*Gn*		*Gn*								*Br*		*Br*
Pb Coat	1	5.0^d	*35R*^{e}		*100R+Y*		*Y*		*5R*		*40PHR*		*40R*								*35SR*		*35SR*
18% Cr–8% Ni	1		*D*^{e}		*G*		*MG*		*M*		*M*		*M*								*MG*		*MG*

[a] Coated before weaving.

[b] Coated after weaving.

[c] Sample lost after 3-yr exposure.

[d] Pinhole rusting previously.

[e] Test discontinued after 10-yr exposure.

* Farm field fence and barbed wire made from these same wire lots corrode at about the same rate as the unfabricated wire, except that in the case of farm field fence the bottom part of the fence corrodes less than the upper part; in the case of barbed wire the barbs show rust a year or two before rust appears on the main wire.

TABLE II.—REPORT OF 1951 AND 1952 INSPECTIONS OF BARBED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate M and G; GY = predominantly gray but showing indication of Y; RB = rust on barbs; SR = superficial rust; asterisk (*) = speckled; R + Y = indistinguishable proportions of R and Y.

Coating Group	Average Coating, oz per sq ft of surface[a]	Wire[b]	State College, Pa.		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			13.42 yr	14.42 yr	15.00 yr	16.00 yr	14.88 yr	15.82 yr	14.93 yr	15.98 yr	14.91 yr	15.90 yr	14.83 yr	15.80 yr	14.83 yr	15.79 yr	14.85 yr	15.80 yr
0.20 to 0.30	0.30	No. C410....	100R	100R	100R	100R	100R	100R	100R	70R	100R	100R	50R	75R	100R	100R	15R	40R
	0.24	No. C411....	100R	100R	100R	100R	100R	100R	30R	25R	100R	100R	75R	90R	100R	100R	25R	50R
	0.29	No. C412....	100R	100R	100R	100R	100R	100R	20R	20R	100R	100R	25R	25R	20R	20R	G	G
0.35 to 0.45	0.37	No. C421....	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	20R	20R	15R	10R	G	G
	0.47	No. C422....	100R	100R	100R	100R	80R	100R	20R	YRB	100R	100R	20R	20R	25R	15R	G	G
	0.31	No. C420....	100R	100R	100R	100R	100R	100R	25R	25R	100R	100R	25R	25R	25R	25R	G	G
0.50 to 0.60	0.48	No. C430....	100R	100R	100R	100R	40R	95R	G	G	100R	100R	20R	20R	15R	Y	G	G
	0.54	No. C431....	100R	100R	100R	100R	10R	10R	G	YRB	75R	75R	25R	25R	20R	YR	G	G
	0.57	No. C432....	GRB	50R	100R	100R	GRB	GRB	MG	MG	100R	100R	G	G	G	G	MG	MG
0.70 to 0.85	0.72	No. C441....	GRB	GRB	100R	100R	GRB	GRB	MG	MG	70R	80R	G	G	G	G	MG	MG
	0.78	No. C442....	GRB	25R	100R	100R	GRB	GRB	MG	MG	30R	50R	G	G	G	G	MG	MG
	0.74	No. C443....	70R	100R	100R	100R	GRB	30R	MG	G	100R	100R	G	G	G	G	MG	MG
0.80 to 1.00	0.97	No. C445....	GY	GY	5R	25R	G	G	M	MG	GY	GY	G	G	G	G	MG	MG
	0.92	No. C446....	G	GRB	20R	60R	G	GRB	MG	MG	10R	10R	G	G	G	G	MG	MG
1.60 to 1.80	1.64	No. C450....	GY	GY	20R	60R	G	G	MG	MG	20R	20R	G	G	G	G	MG	MG
	1.50	No. C452....	G	G	G	95R	G	G	M	MG	MG	GY	G	G	G	G	MG	MG
	1.83	No. C451....	GRB	GRB	20R	40R	GRB	GRB	M	MG	30R	40R	G	G	G	G	MG	MG
Heavy Coating	1.99	No. C494....	G	G	G	G	G	G	MG	MG	MG	MG	G	G	G	G	MG	MG
	1.99	No. C494....	G	G	G	G	G	G	MG	MG	MG	MG	G	G	G	G	MG	MG
	2.00	No. C447....	GRB	GRB	40R	50R	GRB	GRB	M	MG	20R	30R	G	G	G	G	MG	MG
Copper-covered (3.97 oz per sq ft)......		No. C025....	Gn	Gn	Gn	Gn	...	Br	Br	Br	Gn	Gn	BrRB	BrRB	Br	Br	BrRB	BrRB
		No. C025....	Gn	Gn	...	...	Br	...	...	...	...	...	...	...	...	...	...	...
Lead-coated (1.83 oz per sq ft)...........		No. C045....	...	...	50PHR	40PHR	20R	30R	10PHY	10PHY	50R	30R	...	...	3R	4R	25SR	25SR
		No. C045....	...	..	50PHR	40PHR	20R	20R	...	...	50R	30R	35SR	35SR	3R	4R	...	...

[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen opposite:

Barbed Wire	Unfabricated Wire	Barbed Wire	Unfabricated Wire
No. C410	No. 410	No. C441	No. 442
No. C411	...	No. C442	No. 441
No. C412	No. 412	No. C443	No. 443
No. C421	...	No. C445	...
No. C422	No. 421	No. C446	...
No. C420	No. 423	No. C447	...
No. C430	No. 431	No. C450	...
No. C432	No. 432	No. C451	No. 451
No. C431	No. 430	No. C494	

Findings from Weight-Loss Specimens:

At the start of the test, zinc-coated wire specimens were exposed at Pittsburgh, at Sandy Hook, and at State College to determine the rate of loss of coating. These tests have now been completed and summarized in the 1947 report. The over-all average annual loss of coating in ounces per square foot of surface at Pittsburgh is 0.369; at Sandy Hook it is 0.117; and at State College it is 0.060.

Findings from Visual Examination for Corrosion at the Test Plots:

The condensed and summarized inspection records of corrosion through 1952 are shown in Table A. In this table is shown the average number of years until first rust and until 100 per cent rust appeared at various locations on all groups of specimens where progress of rusting has been sufficient to determine the values. For all other groups the average state of corrosion at the last inspection is shown. The galvanized specimens are divided into the coating weight groups established originally in the test to cover galvanized wire supplied under commercial specification at that time. The detailed inspection records collected in 1951 and 1952 are presented in Table I (Plate I) for unfabricated wire, Table II for barbed wire, Table III for farm field fence, Table IV for chain link fence, and Table V for wire strand. These tables are similar in form to the tables in previous reports.

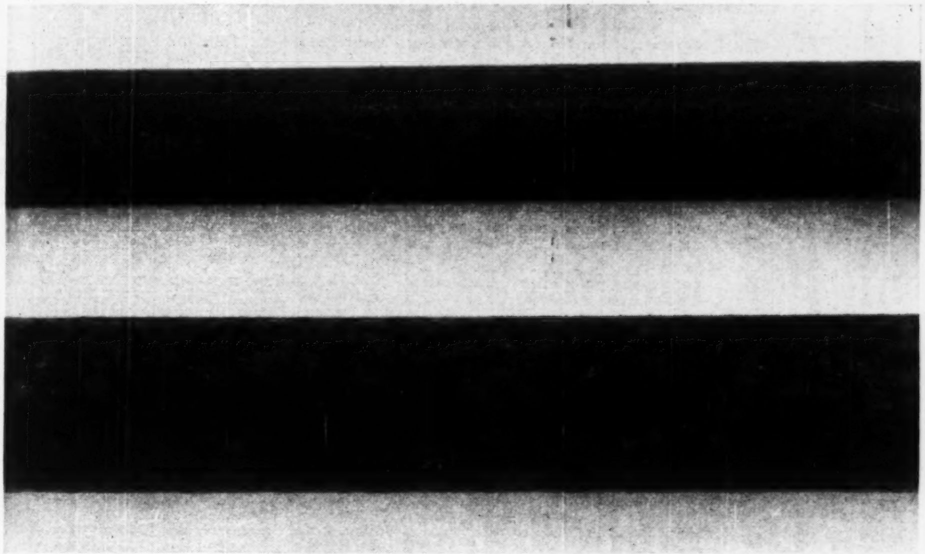

FIG. 1.—Wire Lots Nos. 139 and 247—Galvanized Steel Exposed 15 yr at College Station, Tex. (×5).

The corrosion performance of any individual specimen of barbed wire or of farm field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens listed in Table I (Plate I). The Reference Information Table (Plate II) shows the lot numbers of wires used in fabricating all farm field fence and barbed wire samples.

At the time of inspection, records are

TABLE III.—REPORT OF 1951 AND 1952 INSPECTIONS OF FARM FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR. PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominantely gray but showing indication of Y; SY or SR = superficial Y and superficial R, respectively; asterisk (*) = speckled appearance. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Average Coating, oz per sq ft of Surface[a]	Fence	State College, Pa.[b]		Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Sta., Tex.		Davis, Calif.	
			13.42 yr	14.42 yr	15.00 yr	16.00 yr	14.88 yr	15.82 yr	14.93 yr	15.88 yr	14.91 yr	15.90 yr	14.83 yr	15.80 yr	14.83 yr	15.79 yr	14.85 yr	15.87 yr
0.20 to 0.30 Avg. 0.28	0.27	No. 512	100R	100R	100R	100R	100R	100R	70R	30R	100R	100R	80R	80R	100R	100R	75R	75R
	0.30	No. 410	100R	100R	100R	100R	100R	100R	100R	50R	100R	100R	60R	60R	100R	100R	60R	90R
	0.28	No. 411	100R	100R	100R	100R	100R	100R	20R	5R	100R	100R	35R	60R	60R	80R	20R	60R
	0.29	No. 510	100R	100R	100R	100R	100R	100R	10R	30R	100R	100R	50R	65R	90R	100R	30R	50R
	0.29	No. 412	100R	100R	100R	100R	100R	100R	20R	10R	100R	100R	60R	90R	60R	90R	15R	40R
	0.24	No. 511	100R	100R	100R	100R	100R	100R	50R	20R	100R	100R	90R	95R	95R	98R	80R	100R
0.25 to 0.35 Avg. 0.30	0.25	No. 315	100R	100R	100R	100R	100R	100R	60R	40R	100R	100R	85R	95R	100R	100R	90R	100R
	0.28	No. 215	100R	100R	100R	100R	100R	100R	70R	30R	100R	100R	70R	70R	100R	100R	80R	90R
	0.38	No. 317	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	25R	25R	25R	20R	G	GY
	0.32	No. 316	100R	100R	100R	100R	100R	100R	20R	15R	100R	100R	30R	40R	80R	80R	15R	25R
	0.30	No. 216	100R	100R	100R	100R	100R	100R	10R	10R	100R	100R	20R	25R	30R	30R	GY	15R
0.35 to 0.45 Avg. 0.38	0.36	No. 520	100R	100R	100R	100R	100R	100R	10R	GY			20R	20R	50R	100R	G	GY
	0.35	No. 422	100R	100R	100R	100R	100R	100R	25R	5R	100R	100R	35R	35R	40R	50R	G	15R
	0.31	No. 423	100R	100R	100R	100R	100R	100R	GY	10R	100R	100R	20R	15R	40R	50R	G	GY
	0.44	No. 522	100R	100R	100R	100R	90R	80R	GY	G	100R	100R	15R	15R	20R	25R	G	GY
	0.38	No. 521	100R	100R	100R	100R	100R	100R	10R	10R	100R	100R	40R	50R	50R	55R	5R	20R
	0.47	No. 421	100R	100R	100R	100R	100R	100R	10R	10R	100R	100R	20R	20R	30R	30R	5R	15R
0.45 to 0.55 Avg. 0.46	0.44	No. 325	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	25R	25R	20R	10R	G	GY
	0.50	No. 228	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	35R	35R	15R	15R	G	G
	0.51	No. 227	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	25R	25R	20R	15R	G	G
	0.38	No. 326	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	30R	30R	25R	25R	G	GY
	0.47	No. 327	100R	100R	100R	100R	60R	100R	GY	G	100R	100R	15R	15R	8R	GY	G	GY
	0.45	No. 225	100R	100R	100R	100R	50R	75R	G	G	100R	100R	15R	15R	15R	GY	G	G
0.50 to 0.60 Avg. 0.54	0.61	No. 530	50R	50R	100R	100R	50R	50R	MG	MG	60R	100R	MG	MG	G	G	MG	MG
	0.57	No. 432	GY	40R	100R	100R	G	G	MG	MG	75R	90R	MG	MG	G	G	MG	MG
	0.60	No. 531	100R	100R	100R	100R	50R	50R	MG	MG	95R	99R	MG	MG	G	GY	G	MG
	0.48	No. 431	100R	100R	100R	100R	10R	10R	G	G	100R	100R	15R	10R	20R	GY	G	G
	0.54	No. 430	100R	100R	100R	100R	50R	25R	GY	GY	100R	100R	15R	15R	15R	GY	G	GY
	0.49	No. 532	100R	100R	100R	100R	90R	90R	GY	GY	100R	100R	20R	20R	15R	5R	G	G
	0.49	No. 433	100R	100R	100R	100R	90R	95R	GY	GY	100R	100R	15R	15R	20R	5R	G	G
0.60 to 0.70 Avg. 0.64	0.69	No. 338	30R	15R	30R	55R	G	G	MG	MG	50R	60R	MG	MG	G	G	MG	MG
	0.68	No. 236	G	TrR	75R	100R	G	G	MG	MG	10R	50R	MG	MG	G	G	MG	MG
	0.61	No. 335	50R	100R	100R	100R	20R	50R	MG	MG	90R	100R	G	G	G	G	MG	MG
	0.63	No. 237	80R	90R	90R	95R	25R	75R	MG	MG	90R	95R	MG	MG	GY*	GY*	MG	MG
	0.57	No. 337	100R	100R	100R	100R	5R	5R	GY	GY	100R	100R	20R	20R	10R	10R	G	G
	0.67	No. 235	90R	100R	100R	100R	50R	50R	GY	GY	90R	100R	20R	20R	10R	10R	G	GY
	0.66	No. 336	100R	100R	100R	100R	10R	10R	GY	GY	100R	100R	25R	25R	Y	Y	G	G
	0.60	No. 238	90R	100R	100R	100R	10R	20R	GY	GY	100R	100R	25R	25R	Y	Y	G	G

0.70 to 0.85 Avg. 0.74	0.70	No. 540	GY	G	70R	85R	G	G	MG	MG	10R	90R	MG	MG	G	G	MG	MG
	0.72	No. 442	G	G	10R	40R	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG
	0.79	No. 543	70R	90R	100R	100R	10R	30R	MG	MG	90R	98R	G	G	G	G	MG	MG
	0.78	No. 441	30R	50R	100R	100R	5R	5R	MG	MG	60R	90R	G	G	G	G	MG	MG
	0.70	No. 542	80R	100R	90R	95R	20R	25R	G	MG	80R	95R	G	G	G	G	G	G
	0.74	No. 443	70R	75R	100R	100R	G	5R	MG	MG	95R	100R	MG	MG	G	G	MG	MG
	0.76	No. 541	70R	75R	90R	100R	20R	25R	MG	MG	50R	90R	G	G	G	G	MG	MG
	0.76	No. 440	40R	50R	95R	100R	15R	25R	MG	MG	60R	80R	G	G	G	G	MG	MG
0.80 to 1.00 Avg. 0.90	1.05	No. 348	G	G	5R	60R	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
	0.81	No. 248	G	G	3R	5R	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	0.88	No. 347	GY	TrR	80R	85R	G	5R	MG	G	10R	50R	GY*	GY*	G	G	MG	MG
	0.95	No. 246	GY	TrR	40R	95R	G	10R	MG	MG	5R	20R	G	G	G	G	MG	MG
	0.88	No. 346	10R	20R	100R	95R	G	5R	MG	MG	70R	80R	G	G	G	G	MG	MG
	0.81	No. 245	40R	75R	100R	100R	G	G	MG	MG	75R	90R	MG	MG	G	GY*	MG	MG
	0.84	No. 345	60R	70R	90R	90R	G	G	GY	GY	80R	100R	GY*	GY*	GY*	GY*	G	G
	0.96	No. 247	30R	30R	60R	90R	G	G	GY	GY	50R	60R	GY*	GY*	GY*	GY*	G	G
1.60 to 1.80	1.75	No. 350	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	1.69	No. 252	G	G	G	G	G	G	MG	MG	M	MG	MG	MG	G	G	MG	MG
	1.75	No. 352	G	G	G	G	G	G	MG	MG	GY	GY	G	G	G	G	MG	G
Avg. 2.76	2.80	No. 397	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	2.72	No. 298	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
12–14% Cr, cold drawn		No. 001	MG	MG	M	M	SY	SY	MG*	MG*	M*	MG*	80SR	80SR	25R	20R	55SR	50R
12–14% Cr, air quenched		No. 002	MG	MG	M	M	SY	SY	M	MG*	M*	MG*	40SR	45SR	10R	8R	30SR	30SR
18% Cr, 8% Ni		No. 003	M	M	M	M	M	M	M	M	M	M	MG	MG	MSY*	M*	MG	MG
Copper-covered (7.42 oz per sq ft)		No. 020	Gn	Gn	Gn	Gn	Br	Br	Br	Br	Gn	Gn	Br	Br	Br	Br	Br	Br
Lead-coated (1.48 oz per sq ft)		No. 040	100Y+R	100R+Y	30PHR	35PHR	30R	30R	25R	15R	40R	40R	40SR	40SR	15R	20R	40R	40R

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

[b] The rating for the state of corrosion is based on the appearance of the top half of the fence. At all locations the bottom part of the fence shows less corrosion than the top part.

TABLE IV.—REPORT OF 1951 AND 1952 INSPECTIONS OF CHAIN LINK FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y; R+Y = indistinguishable proportions of R and Y; SR = superficial rust. Figures (as 5, 20, 50, 100) are percentages.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire 0.128 in. diam.

Zinc Coating, oz per sq ft of Surface, by Stripping Test	Fence	Sandy Hook, N. J.*[c]	Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ithaca, N. Y.		Santa Cruz, Calif.		Davis, Calif.	
		14.44 yr	14.45 yr	15.45 yr	13.42 yr	14.42 yr	15.00 yr	16.00 yr	14.91 yr	15.90 yr	14.83 yr	15.80 yr	14.35 yr	15.87 yr
0.29	No. E280[a]	100R	100R	100R	100R	100R	100R	100R	100R	100R	50R	50R	25R	50R
0.52	No. E281	100R	100R	100R	80R	100R	100R	100R	90R	100R	10R	10R	5R	5R
0.37	No. E282	100R	100R	100R	100R	100R	100R	100R	100R	100R	10R	10R	G	3R
1.22	No. E185	100R	100R	100R	Y	Y	5R	20R	GY	GY	MG	MG	MG	MG
1.13	No. E285	100R	100R	100R	5R	5R	75R	80R	GY	GY	MG	MG	G	MG
2.92	No. E290	G	GY	GY	G	G	G	G	M	MG	MG	MG	MG	MG
2.09	No. E291	2R	GY	GY	G	G	G	G	MG	MG	MG	MG	MG	MG
1.60	No. H280[a]	100R	95R	100R	G	TrR	40R	75R	10R	10R	G	G	MG	MG
1.83	No. H185	100R	95R	100R	GY	GY	GY	10R	GY	Y	MG	MG	MG	MG
1.60	No. H186	100R	98R	100R	G	GY	GY	20R	GY	Y	G	G	MG	MG
1.61	No. H187	100R	95R	100R	G	GY	GY	10R	GY	Y	G	G	MG	MG
1.55	No. H188	100R	100R	100R	GY	GY	20R	60R	GY	Y	MG	MG	MG	G
1.36	No. H285	100R	90R	100R	GY	GY	GY	30R	GY	Y	G	G	G	MG
1.97	No. H286	100R	95R	100R	GY	GY	GY	25R	GY	Y	MG	MG	MG	MG
1.46	No. H287	100R	90R	100R	G	GY	5R	40R	5R	5R	MG	MG	MG	MG
1.74	No. H288	100R	98R	100R	G	GY	GY	10R	GY	Y	G	G	G	MG
1.40	No. H289[b]	90R	100R	100R	G	GY	G	G	MG	MG	MG	MG	MG	MG
4.21	No. H290	GY	GY	Y	G	G	GY	GY	GY	GY	MG	MG	MG	MG
Lead-coated L050 (1.75 oz per sq ft)		100R+Y	Y	Y	100R+Y	5R	100R+Y	40PHR	60R	40R	35SR	35SR	35SR	35SR
Copper-covered K030 (8.52 oz per sq ft)		Gn	Gn	Gn	Gn	Gn	Gn	Gn	Br	Gn	Br	Br	Br	Br
Corrosion-resistant steel J011		G	MG	MG	MG	M	M	M	M	M	MG	MG	MG	MG

[a] E numbers, coating applied before weaving. H numbers, coating applied after weaving.

[b] While this sample has an H number, its appearance indicates that it was galvanized before weaving.

* Specimens destroyed shortly after this inspection.

[c] The rating for the state of corrosion is based on the appearance of the fence at eye level. At all exposure sites the bottom part of the fence is less corroded than the upper part of the fence.

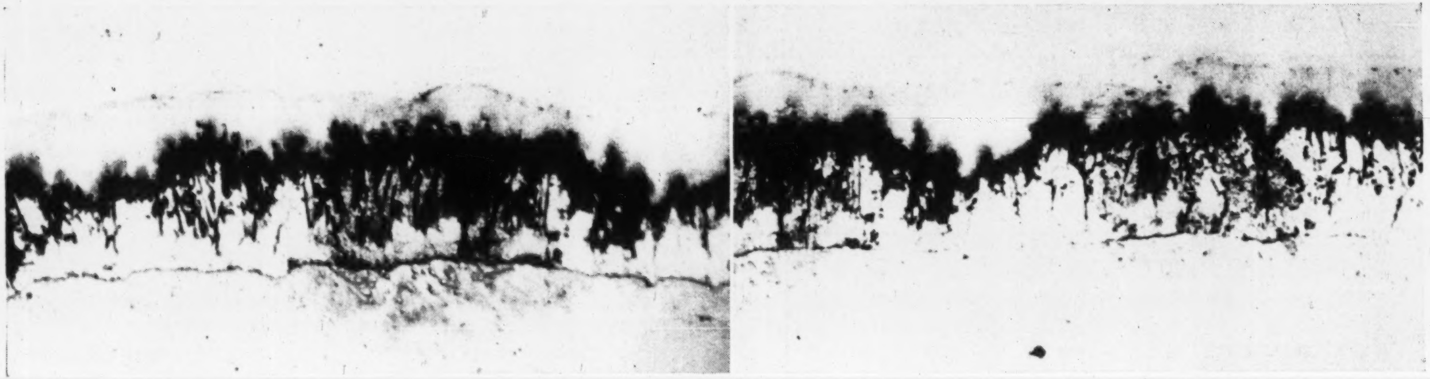
TABLE V.—REPORT OF 1951 AND 1952 INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y; R+Y = indistinguishable proportions of R and Y.

Zinc Coating, oz per sq ft of surface	Strand	Sandy Hook, N. J.		Bridgeport, Conn.		State College, Pa.		Lafayette, Ind.		Ames, Iowa	
		14.44 yr	15.45* yr	14.45 yr	15.45 yr	14.30 yr	15.30 yr	15.00 yr	16.00 yr	14.88 yr	15.82 yr
0.69	No. D345..........	45R	85R	90R	100R	G	G	GY	5R	G	G
0.88	No. D346..........	50R	50R	35R	100R	GY	GY	GY	10R	G	G
0.87	No. D347..........	50R	50R	20R	75R	GY	GY	10R	40R	G	G
0.93	No. D348..........	50R	80R	85R	100R	G	G	5R	10R	G	G
1.10	No. D360..........	G	25R	GY	GY	G	G	G	G	G	G
1.17	No. D361..........	10R	40R	15R	60R	GY	GY	GY	10R	G	G
1.03	No. D362..........	50R	60R	65R	60R	GY	GY	GY	30R	G	G
1.51	No. D365..........	GY	5R	GY	Y	GY	GY	GY	25R	G	G
1.39	No. D366..........	G	20R	GY	GY	G	G	G	G	G	G
1.32	No. D367..........	10R	50R	35R	30R	GY	GY	Y	30R	G	G
1.71	No. D371..........	G	G	GY	G	G	G	G	G	G	G
2.47	No. D375..........	G	G	GY	G	G	G	G	G	G	G
Lead Coating (1.96 oz per sq ft)......	No. D045..........	100R+Y	100R+Y	5R	20R	100R+Y	10R	25PHR	50PHR	40R	60R

Zinc Coating, oz per sq ft of surface	Strand	Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
		14.93 yr	15.88 yr	14.91 yr	15.90 yr	14.83 yr	15.80 yr	14.83 yr	15.79 yr	14.85 yr	15.87 yr
0.69	No. D345..........	MG	MG	3R	13R	G	G	G	G	G	G
0.88	No. D346..........	MG	MG	GY	GY	MG	MG	G	G	MG	MG
0.87	No. D347..........	MG	GY	GY	GY	G	G	G	G	G	G
0.93	No. D348..........	MG	MG	MG	1R	MG	MG	G	G	G	G
1.10	No. D360..........	MG	MG	MG	MG	MG	MG	G	G	MG	MG
1.17	No. D361..........	MG	MG	GY	Y	G	G	G	G	G	G
1.03	No. D362..........	MG	MG	GY	GY	MG	MG	G	G	G	G
1.51	No. D365..........	MG	GY	GY	Y	G	G	G	G	G	G
1.39	No. D366..........	MG	MG	MG	G	MG	MG	G	G	G	G
1.32	No. D367..........	MG	MG	GY	3R	MG	MG	G	G	G	G
1.71	No. D371..........	MG	MG	MG	G	MG	MG	G	G	MG	MG
2.47	No. D375..........	MG	MG	MG	G	MG	G	G	G	G	G
Lead Coating (1.96 oz per sq ft)......	No. D045..........	25R	25R	50R	70R	60SR	60SR	10R	10PHR	10R	10R

* Specimens destroyed shortly after this inspection.



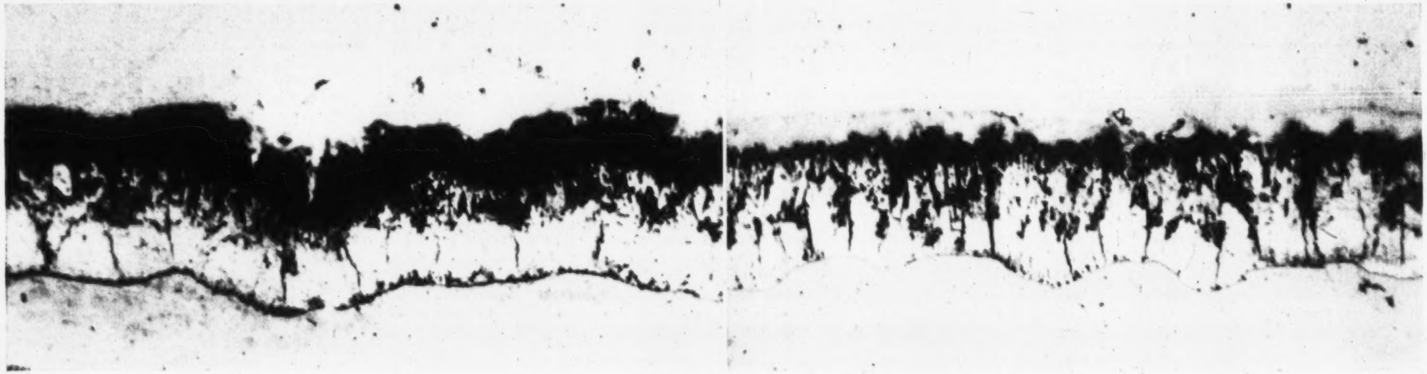
College St ation, Tex.

Santa Cruz, Calif.

Wire Lot No. 541

White layers at top are nickel plated, including a stained layer in immediate contact with coating. Black irregular layer is corrosion products and white mottled layer beneath is remaining galvanized coating. Gray area at bottom is steel base metal.

Relative amount of corrosion product (black irregular layer) over zinc-iron alloy coating is indicated to be less on this sample than on the Texan sample.



Wire Lot No. 138

Thickness of black outside layer much heavier on this wire indicated more corrosion products on the surface of this wire.

Corrosion product again is appreciably less than on samples exposed at College Station, Texas.

FIG. 2.—Wires Exposed Approximately 15 yr at College Station, Tex., and Santa Cruz, Calif. (Uneteched, tranverse sections ×750).

TABLE VI.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b]—SANDY HOOK—500 SERIES (14½ GAGE); 400 SERIES (12½ GAGE); 300 SERIES (11 GAGE); 200 SERIES (9 GAGE); AND 100 SERIES (6 GAGE) UNFABRICATED WIRES.

TABLE VI.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b]—SANDY HOOK—500 SERIES (14½ GAGE); 400 SERIES (12½ GAGE); 300 SERIES (11 GAGE); 200 SERIES (9 GAGE); AND 100 SERIES (6 GAGE) UNFABRICATED WIRES.

Wire Lot	Original Breaking Load, lb	Exposure Until First Rust, yr	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
			Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss	
				%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr
No. *501[a]	500	0	1.44	22.0	15	1.90	24.0	13	2.42	25.0	10	2.90	32.0	11	3.45	36.0	11	5.45	54.0	10	6.90	58.0	9
No. *506	520	0	1.44	25.0	17	1.90	25.0	13	2.42	27.9	11	2.90	32.8	11	3.45	36.5	11	5.45	55.8	10	6.90	56.8	9
No. *507	590	0	1.44	22.7	16	1.90	26.0	14	2.42	31.1	13	2.90	36.2	12	3.45	42.0	12	5.45	63.0	12	6.90	68.9	10
No. 510	500	1.75	0	2.0	0	0.15	−2.0	0	1.70	16.0	9	3.70	28.0	8	5.15	41.1	8	6.70	49.0	7	8.60	60.0	7
No 511	280	1.75	0	12.0	0	0.15	−1.7	0	1.70	7.0	4	3.70	33.3	9	5.15	47.4	9	6.70	59.6	9	8.60	65.0	8
No. 512	320	1.55	0	10.7	0	0.35	4.5	13	1.90	20.0	11	3.90	35.4	9	5.35	41.8	9	6.90	55.4	8	8.80	63.0	7
No. 520	280	2.70	0	3.5	0	1.75	8.9	5	3.75	23.2	6	5.20	33.9	7	5.75	41.1	7	7.65	44.6	6	9.70	57.2	6
No. 521	270	2.90	0	3.5	0	1.55	0	0	3.55	20.4	6	5.00	31.5	6	5.55	46.3	8	7.45	55.6	8	9.50	66.7	7
No. 522	500	2.90	0	2.0	0	1.55	7.0	5	3.55	26.0	7	5.00	37.0	7	5.55	48.0	9	7.45	57.0	8	9.50	68.0	7
No. 530	310	2.90	3.55	3.2	1	5.00	24.2	5	5.55	29.0	5	6.50	35.5	5	7.45	42.0	6	9.50	40.3	4	11.55	71.0	6
No. 531	500	4.70	1.75	9.0	5	3.20	27.0	8	3.75	33.0	9	4.70	45.0	7	5.65	47.0	8	7.70	70.0	9	9.75	83.0	8
No. 540	300	5.90	2.55	18.3	7	3.50	21.7	6	5.50	42.0	8	8.55	73.5	9	9.10	66.7	7						
No. 541	350	5.40	3.05	25.7	8	4.00	34.3	9	6.00	52.2	9	9.05	72.9	8	9.60	70.0	7						
No. 542	510	5.50	2.95	35.2	12	3.90	34.3	9	5.90	54.5	9	8.95	77.5	9	9.50	72.0	8						
No. 543	300	5.90	2.55	16.7	6	3.50	18.3	5	5.50	44.7	8	9.75	75.0	8	9.10	66.7	7						
No. *400[a]	470	0	1.44	22.0	13	1.90	14.5	8	2.42	15.6	6	3.90	26.0	7	5.45	36.5	7	6.90	37.7	5	11.43	53.8	5
No. *401[a]	440	0	1.44	18.7	12	1.90	15.5	8	2.42	16.7	7	3.90	30.0	8	5.45	37.8	7	6.90	44.4	6	11.43	60.7	5
No. *405	470	0	1.44	19.6	14	1.90	15.5	8	2.42	15.5	6	3.90	29.9	8	5.45	41.2	8	6.90	42.3	6	11.43	55.4	5
No. *406	460	0	1.44	19.0	13	1.90	15.0	8	2.42	14.9	6	3.90	25.5	7	5.45	35.1	6	6.90	34.0	5	11.43	45.6	4
No. 410	570	1.80	0	3.5	0	1.65	5.3	3	3.10	21.9	7	3.65	18.4	5	5.10	25.5	5	8.55	41.0	5	10.60	43.8	4
No. 411	615	1.85	0	−5.7	0	1.60	4.9	3	3.05	11.4	4	3.60	14.6	4	5.05	21.9	4	8.50	37.4	4	10.60	41.4	4
No. 412	830	2.50	0	5.0	0	0.95	6.0	6	2.40	...	...	2.95	...	...	4.40	20.5	5	7.85	35.0	4	10.00	42.2	4
No. 421	470	2.80	0	6.5	0	1.65	4.3	3	3.65	12.8	4	5.10	25.5	5	7.55	35.0	5	9.60	43.7	5	11.65	60.7	5
No. 422	500	2.90	0	6.0	0	1.55	4.0	3	3.55	13.0	4	5.00	19.0	4	7.45	27.0	4	9.50	34.0	4	11.55	49.0	4
No. 423	400	2.75	0	7.5	0	1.70	5.0	3	3.70	12.5	3	...	...	...	7.60	35.0	5	9.70	41.3	4	...	...	...
No. 430	780	4.40	2.05	4.5	2	3.50	21.2	6	4.05	20.5	5	5.00	25.7	5	5.95	30.8	5	8.00	42.3	5	10.05	55.2	5
No. 431	880	3.70	2.75	13.1	5	4.20	22.7	5	4.75	24.4	5	5.70	30.0	6	6.65	30.6	5	8.70	39.2	4	10.75	54.0	5
No. 432	510	6.50	0	−10.0	0	1.40	12.7	9	1.95	10.8	5	2.90	16.0	5	3.85	18.6	5	6.00	28.4	5	7.95	43.2	5
No. 440	590	4.70	3.75	26.3	7	4.70	28.0	6	6.70	33.9	5	9.75	55.1	6									
No. 441	520	6.50	1.95	11.5	6	2.90	18.3	6	4.90	30.4	6	7.95	50.0	6									
No. 442	470	7.40	1.05	19.2	18	2.00	9.6	5	4.00	25.9	6	7.05	48.0	7									
No. 443	750	5.90	2.55	18.0	7	3.50	20.7	6	5.50	27.3	5	8.55	58.0	7									
No. 444	570	5.70	2.75	18.4	7	3.70	17.5	5	5.70	25.6	5	8.75	46.5	5									
No. 495	750	5.90	2.55	17.3	7	3.50	18.0	5	5.50	23.1	4	8.55	47.3	6									

(Continued on pp. 112 and 113)

TABLE VI.—*Concluded.*

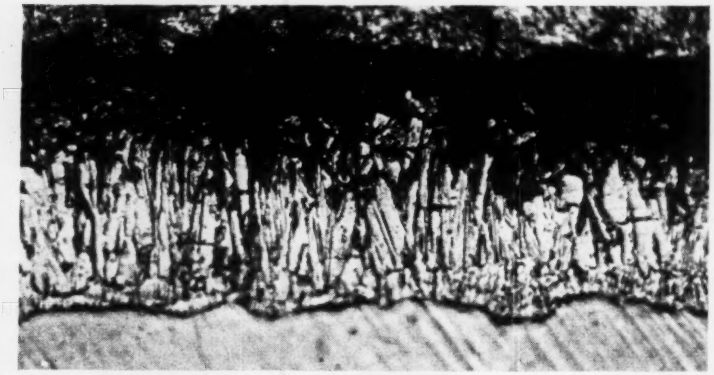
Wire Lot	Original Breaking Load, lb	Exposure Until First Rust, yr	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
			Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss	
				%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr
No. *300[a]	730	0	1.45	13.5	9	1.90	12.0	6	3.45	16.9	5	3.90	20.3	5	5.95	22.3	4	14.45	53.5	4			
No. *302[a]	1100	0	1.45	19.3	13	1.90	16.3	9	3.45	21.8	6	3.90	24.5	6	5.95	26.4	4	14.45	52.3	4			
No. *305	1070	0	1.45	12.5	9	1.90	14.8	8	2.90	17.0	6	3.90	18.9	5	5.95	26.3	4	14.45	50.7	4			
No. *306	700	0	1.45	14.0	10	1.90	12.0	6	2.90	11.3	4	3.90	16.9	4	5.95	20.4	3	14.45	44.4	3			
No. 315	900	1.60	...	−1.0	...	3.30	12.8	4	4.85	12.2	3	8.70	34.7	4	12.85	43.9	3						
No. 316	1035	2.70	...	−8.5	...	2.20	1.5	...	3.75	6.8	2	7.70	22.6	3	11.75	35.8	3						
No. 317	795	3.75		−2.0	...	1.15	2.5	2	2.70	10.7	4	9.80	26.6	3	10.70	40.3	4						
No. 325	730	3.80	2.65	4.8	2	4.65	13.0	3	5.60	13.7	2	7.60	19.2	3	10.60	33.6	3						
No. 326[a]	700	3.85	2.60	12.9	5	4.60	17.1	4	5.65	22.2	4	7.65	26.9	3	10.65	42.2	4						
No. 327	1160	3.80	2.65	10.3	4	4.65	22.4	5	5.60	23.3	4	7.60	27.3	4	10.60	40.1	4						
No. 329	720	3.80	2.65	6.3	2	4.65	11.1	2	5.60	19.5	3	7.60	27.4	4	10.60	41.7	4						
No. 335	1040	4.90	3.55	17.8	5	4.50	19.3	4	6.50	25.0	4	9.55	37.1	4									
No. 336	760	4.80	3.65	14.5	4	4.60	21.1	5	6.60	27.7	4	9.65	40.2	4									
No. 337	860	4.80	3.65	19.8	5	4.60	22.7	5	6.60	21.7	3	9.65	40.2	4									
No. 338	1020	7.90	0.55	5.4	10	1.50	9.3	6	3.50	12.8	4	6.55	32.3	5									
No. 345	890	5.90	2.55	13.5	5	3.50	22.5	6	5.50	25.3	5												
No. 346	1070	6.50	1.95	14.0	7	2.90	16.3	6	4.90	18.7	4												
No. 347	860	7.80	0.65	3.5	5	1.60	13.4	8	3.60	16.4	5												
No. 348	980	10.50	...	2.0	...	...	3.6	...	1.00	5.1	5												
No. *200[a]	1010	0	1.45	14.8	10	2.40	13.4	6	3.45	15.8	5	5.95	21.5	4	14.45	47.8	3						
No. *202[a]	1640	0	1.45	8.5	6	2.40	11.0	5	3.45	9.5	3	5.95	15.8	3	14.45	38.1	3						
No. *205	1110	0	1.45	12.5	9	2.40	11.3	5	3.45	14.4	4	5.95	19.8	3	14.45	43.7	3						
No. *206	1480	0	1.45	13.5	9	2.40	10.8	5	3.45	14.9	4	5.95	18.6	3	14.45	40.2	3						
No. 215	1210	1.80	...	1.5	...	3.10	8.7	3	4.65	11.6	2	8.70	26.9	3									
No. 216	1630	2.50	...	1.6	...	2.30	8.0	3	3.95	10.4	3	10.00	24.5	2									
No. 217	1080	2.80	...	−7.0	...	2.15	4.2	2	3.65	0.5	...	9.60	22.2	2	12.50	25.8	2						
No. 282	1320	2.80	0.20	−2.0	10	1.65	10.2	6	3.65	8.7	2	5.10	10.6	2	7.55	20.8	3	11.65	31.4	3			
No. 225	1650	3.90	2.55	7.0	3	4.55	13.0	3	5.50	15.7	3	10.50	30.0	3									
No. 226	1150	3.90	2.55	3.0	1	4.55	10.0	2	5.50	11.7	2	10.05	32.0	3									
No. 227	1640	4.40	2.05	8.3	4	4.05	14.3	3	5.00	17.1	3	10.55	32.0	3									
No. 228	1080	2.90	3.55	3.7	1	5.55	11.1	2	6.50	11.6	2	11.55	23.1	2									
No. 235	1680	5.50	2.95	28.0	10	3.90	29.2	7	5.90	31.1	5	8.95	40.5	4									
No. 236	1270	7.70	0.75	...	...	1.70	...	...	3.70	...	...	6.75	16.1	2	7.3	14.7	2						
No. 237	1540	4.40	4.05	7.5	2	5.00	13.0	3	7.00	16.1	2	10.05	21.2	2									
No. 238	1650	4.80	3.65	13.0	4	4.60	14.8	3	6.60	21.2	3	9.65	26.7	3									
No. 239	1120	5.50	2.95	7.6	3	3.90	8.5	2	5.90	12.9	2	8.95	21.0	2									

No. 245	1570	5.90	2.55	7.0	3	3.50	1.0	...	9.10	22.6	2												
No. 246	1190	7.90	0.55	...	...	1.50	0.3	...	7.10	16.7	2												
No. 247	1790	6.50	1.95	1.6	1	2.90	2.2	1															
No. 248	1450	7.40	1.05	5.2	5	2.00	1.0	...	7.6	23.3	3												
No. 296	1570	5.90	2.55	7.0	3	3.50	1.0	...															
No. 801	1820	8.90	0.50	5.4	10	5.55	30.2	6															
No. *101[a]	2330	0	1.45	7.5	5	2.40	5.4	2	3.45	11.7	3	6.45	15.1	2	14.45	32.2	2						
No. *105[a]	2750	0	1.45	9.5	7	2.40	8.7	4	3.45	12.1	4	6.45	15.6	2	14.45	29.0	2						
No. *102	2110	0	1.45	11.5	8	2.40	12.2	5	3.45	13.1	4	6.45	17.4	3	11.45	31.5	3						
No. *106	2430	0	1.45	5.4	4	2.40	6.9	3	3.45	10.8	3	6.45	8.5	1	11.45	27.7	2						
No. 115	2715	1.75	...	2.5	...	3.15	7.5	2	4.70	8.5	2												
No. 117	2215	2.50		0.5	...	2.40	5.4	2	3.95	6.1	2												
No. 125	1840	3.45	3.00	5.4	2	6.00	10.1	2	11.00	20.4	2												
No. 127[a]	2540	3.90	2.55	1.4	...	5.50	9.2	2	10.55	19.4	2												
No. 135	2660	5.90	2.55	7.5	2	3.50	10.1	3	8.55	18.6	2												
No. 138	1960	5.50	2.95	8.2	3	3.90	7.4	2	8.95	15.6	2												
No. 139	1920	4.80	3.65	9.1	3	4.60	8.6	2	9.65	18.5	2												
No. 148	2550	6.90	1.55	2.7	2	2.50	5.7	2															
No. 149	1910	6.80	1.65	2.1	1	2.60	4.2	2															
No. 800	2670	10.20	...	7.4	...	4.25	11.4	3															

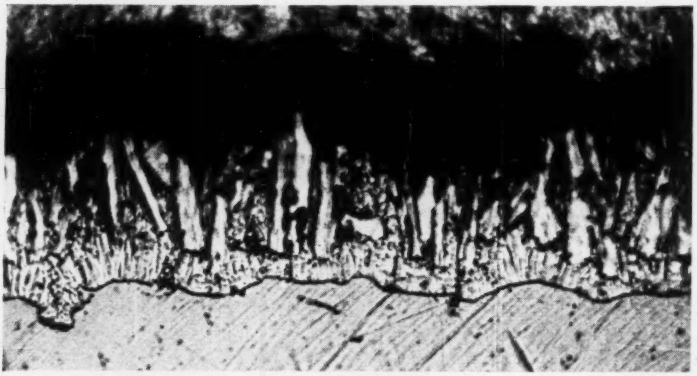
* Uncoated Wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent copper. All other wires have approximately 0.25 per cent copper.

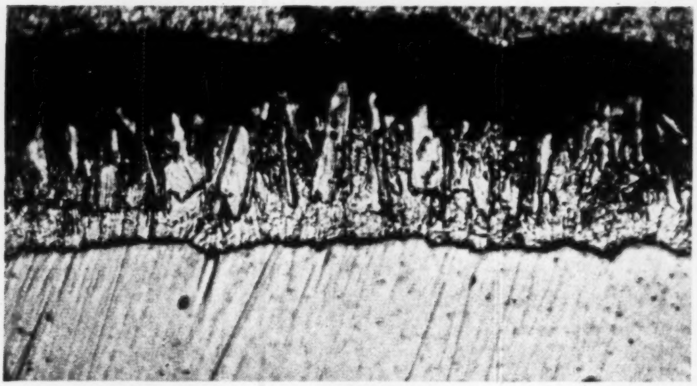
[b] Summarizes data through 1952.



Wire Lot No. 235

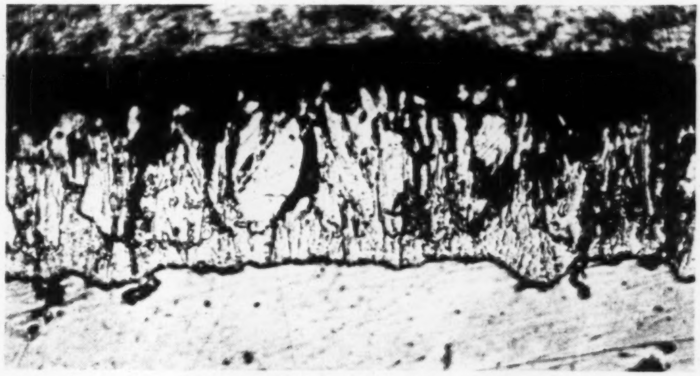


Wire Lot No. 247

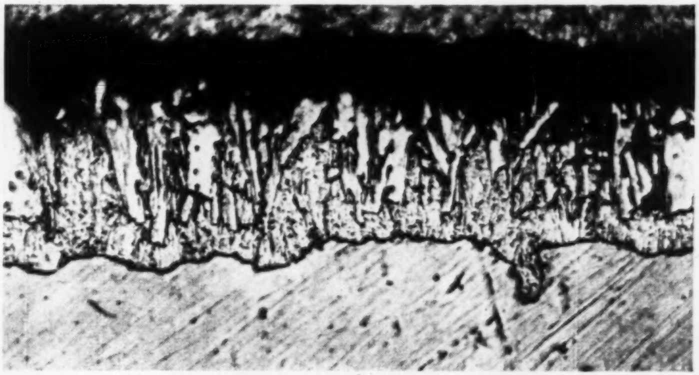


Wire Lot No. 345

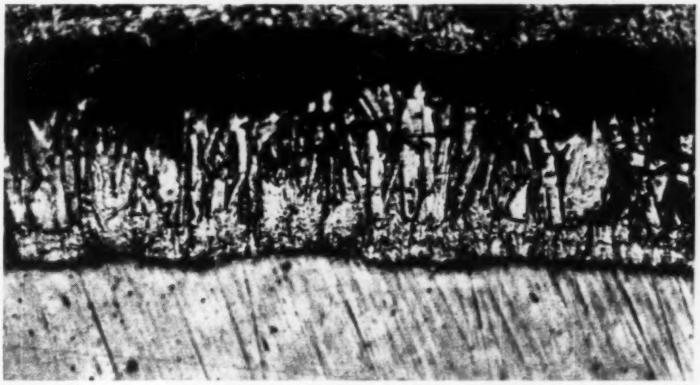
FIG. 3.—Galvanized Steel Wires After 15-yr



Wire Lot No. 440

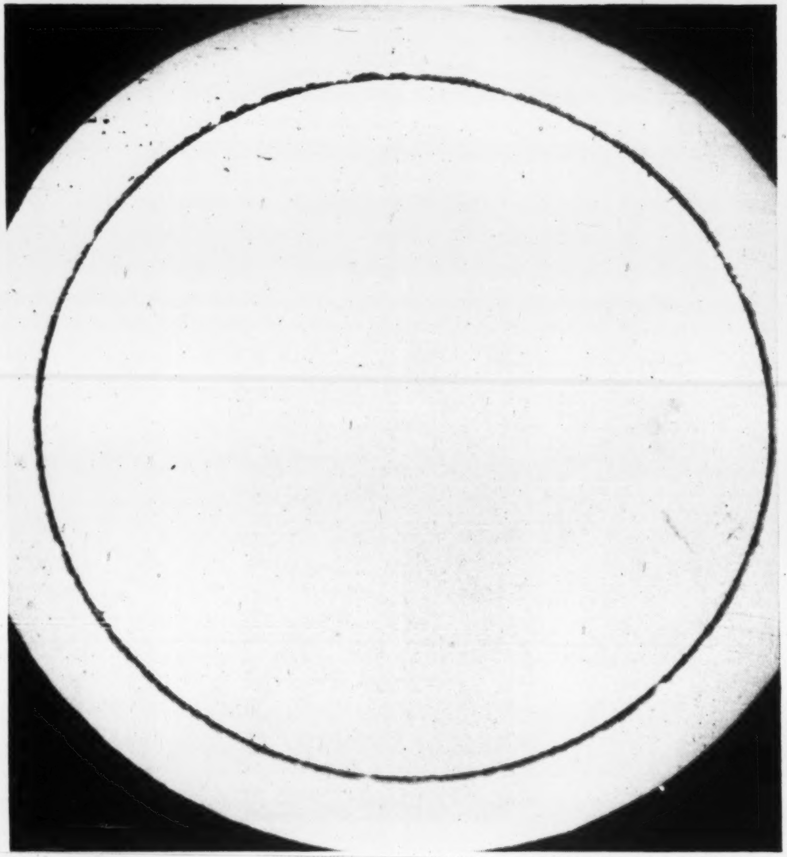


Wire Lot No. 444



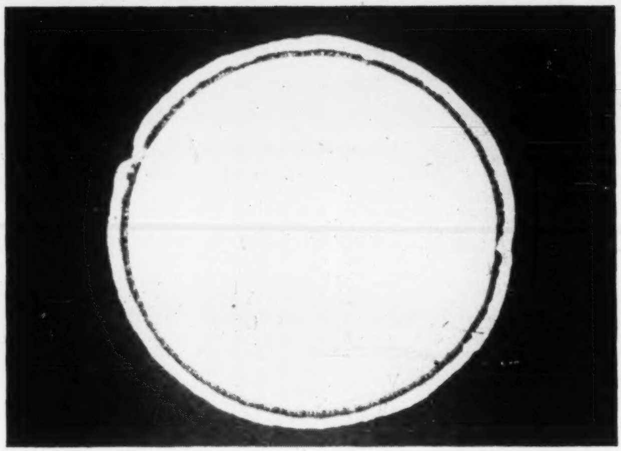
Wire Lot No. 541

Exposure at College Station, Tex. (×750).



Wire Lot No. 235

Photograph illustrating continuity of coating on 0.148-in. wire exposed at College Station, Tex. A few pits are also present in this coating.



Wire Lot No. 138

Photograph illustrating continuity of coating on this sample of 0.076-in. wire exposed at College Station, Tex. Outer layer is nickel plating and intermediate dark or mottled layer is weathered galvanized coating. Note occasional absence of galvanized coating in spots.

Fig. 4.—Continuity of Coating on Wires from College Station, Tex. (Unetched transverse sections).

TABLE VII.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b]—BRIDGEPORT, CONN.—500 SERIES (14½ GAGE); 40 SERIES (12½ GAGE); 300 SERIES (11 GAGE); 200 SERIES (9 GAGE); AND 100 SERIES (6 GAGE) UNFABRICATED WIRES.

Wire Lot	Original Breaking Load, lb	Exposure Until First Rust, yr	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
			Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr	Exp. Since 1R, yr	Tensile Strength Loss, %	Tensile Strength Loss, %/yr
No. *501[a]	500	0	1.45	16.0	11	1.91	23.0	12	2.42	21.0	9	2.91	24.0	8	5.45	33.0	6	8.45	44.0	5	10.35	46.0	4
No. *506	520	0	1.45	21.2	15	1.91	17.4	9	2.42	22.1	9	3.45	25.0	7	5.45	33.6	6	8.45	45.2	5	10.35	46.0	4
No. *507	590	0	1.45	17.6	12	1.91	17.6	9	2.42	20.2	8	3.45	26.9	8	5.45	36.1	6	8.45	48.6	6	10.35	48.0	5
No. 510	500	2.50	0	−2.0	0	0.06	−6.0	0	1.60	5.0	3	3.44	16.0	5	5.95	28.0	5	7.85	36.0	5	10.00	41.0	4
No. 511	280	1.80	0	8.8	0	0.11	−0.4	0	1.65	5.3	3	4.14	19.3	5	6.65	35.1	5	8.55	40.4	5	10.70	43.8	4
No. 512	320	1.65	0	7.6	0	0.26	1.7	0	1.80	13.8	8	4.29	20.0	5	6.85	35.4	5	8.75	38.4	4	10.90	44.7	4
No. 520	280	2.50	1.95	0	0	3.95	12.5	3	5.95	21.4	4	6.90	16.1	2	7.85	23.2	3	10.00	25.0	3			
No. 521	270	3.45	1.00	−3.0	0	3.00	3.7	1	5.05	25.9	5	6.00	22.2	4	6.95	27.8	4	9.10	33.3	4			
No. 522	500	3.45	1.00	1.0	0	3.00	14.0	5	5.05	32.0	6	6.00	34.0	6	6.95	32.0	5	9.10	36.0	4			
No. 530	310	5.40	1.05	12.9	12	2.50	19.3	8	3.05	25.2	8	4.00	27.5	7	6.00	30.3	5	7.00	41.0	6			
No. 531	500	4.70	1.75	12.0	7	3.20	23.0	7	3.75	25.0	7	4.70	34.0	7	6.70	35.2	5	7.70	38.0	5			
No. 540	300	6.20	2.25	16.7	7	3.20	18.4	6	5.20	28.7	6	6.20	25.0	4									
No. 541	350	4.90	3.55	7.1	2	4.50	14.3	3	6.50	24.9	4	7.50	27.1	4									
No. 542	510	5.50	2.95	23.6	8	3.90	30.4	8	5.90	36.3	6	7.00	33.3	5									
No. 543	300	5.90	2.55	13.3	5	3.50	16.7	5	5.50	31.3	6	6.50	26.6	4									
No. *400[a]	470	0	1.45	16.5	12	1.91	10.5	6	3.91	13.5	4	5.45	19.8	4	9.42	28.1	3	12.50	27.1	2			
No. *401[a]	470	0	1.45	15.5	11	1.91	11.0	6	3.91	17.8	5	5.45	24.4	4	9.42	30.0	3	12.50	33.3	3			
No. *405	470	0	1.45	17.5	12	1.91	13.5	7	2.91	13.4	5	5.45	20.6	4	9.42	25.8	3	12.50	33.0	3			
No. *406	460	0	1.45	15.0	11	1.91	8.5	4	2.91	10.7	4	5.45	18.1	3	9.42	21.3	2	12.50	23.4	2			
No. 410	570	1.80	0	3.5	0	0.11	−5.3	0	1.65	3.5	2	4.14	10.5	3	9.60	26.7	3	10.60	22.8	2			
No. 411	615	1.90	0	2.5	0	0	−11.4	0	1.55	1.6	1	4.04	5.7	1	9.10	19.2	2	10.10	22.0	2			
No. 412	830	2.50	0	0	0	0	0	0	1.00	7.2	7	3.44	...	...	8.90	24.3	3	10.00	22.9	2			
No. 421	470	2.70	1.75	3.2	2	3.75	9.6	3	5.75	11.7	2	7.65	20.2	3	9.80	20.2	2	11.75	29.8	2			
No. 422	500	2.70	1.75	5.0	3	3.75	7.0	2	5.75	11.0	2	7.65	17.0	2	9.80	18.0	2	11.75	27.0	2			
No. 423	400	2.50	1.95	2.5	1	3.95	8.7	2	5.95	12.5	2	7.85	18.8	2	10.00	17.5	2	11.95	18.7	2			
No. 430	780	4.90	1.55	...	...	3.00	8.3	3	3.65	8.3	2	5.55	14.1	3	9.65	26.2	3						
No. 431	880	3.50	2.95	10.2	3	4.40	18.7	4	4.95	14.8	3	6.85	22.2	3	10.95	33.6	3						
No. 432	510	5.40	1.05	0	0	2.50	8.8	4	3.05	4.9	2	4.95	15.7	3	9.05	26.5	3						
No. 440	590	4.80	3.65	11.0	3	4.60	20.3	5	6.60	15.8	2	9.65	22.9	2									
No. 441	520	5.90	2.55	8.6	3	3.50	18.3	5	5.50	21.7	4	8.55	28.0	3									
No. 442	470	6.30	2.15	12.8	6	3.10	20.2	6	5.10	21.9	4	8.15	24.6	3									
No. 443	750	5.80	2.65	12.0	5	3.60	18.6	5	5.60	18.2	3	8.65	26.0	3									
No. 444	570	5.60	2.85	2.6	1	3.80	4.4	1	5.80	19.3	3	8.85	16.6	2									
No. 495	750	5.90	2.55	12.0	5	3.50	18.7	5	5.50	18.0	3	8.55	27.4	3									

[Continued on pp. 118 and 119]

TABLE VII.—*Concluded.*

Wire Lot	Original Breaking Load, lb	Exposure Until First Rust, yr	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
			Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr	Tensile Strength Loss %	Tensile Strength Loss %/yr	Exp. Since 1R, yr.	Tensile Strength Loss %	Tensile Strength Loss %/yr
No. *300[a]	770	0	1.45	11.0	8	2.40	8.8	4	3.90	10.8	3	5.95	10.8	2	14.45	25.0	2						
No. *302[a]	1100	0	1.45	17.3	12	2.40	18.2	7	3.90	20.5	5	5.95	19.1	3	14.45	32.3	2						
No. *305	1070	0	1.45	9.0	6	2.40	10.1	4	3.90	15.2	4	5.95	14.7	2	14.45	29.1	2						
No. *306	700	0	1.45	7.0	5	2.40	7.7	3	3.90	7.7	2	5.95	8.5	1	14.45	24.0	2						
No. 315	900	1.70	...	0.0	...	2.75	5.5	2	4.75	9.5	2	8.65	16.1	2	12.75	22.3	2						
No. 316	1035	2.50	...	−3.5	...	1.65	−1.5	...	3.95	1.0	...	7.85	7.7	1	11.65	16.4	1						
No. 317	795	2.80	...	−3.1	...	1.65	0.6	...	3.65	10.7	3	7.55	16.3	2	11.65	25.1	2						
No. 325	730	3.90	2.55	2.1	1	4.55	9.6	2	6.45	10.3		10.55	17.8	2									
No. 326[a]	700	2.90	3.55	8.6	2	5.55	14.6	3	7.45	20.7	3	11.55	27.2	2									
No. 327	1160	3.90	2.55	8.2	3	4.55	13.8	3	6.45	16.8	2	10.55	23.3	2									
No. 329	720	3.80	2.65	4.2	2	4.65	8.3	2	6.55	14.6	2	10.65	20.2	2									
No. 335	1040	5.40	3.05	12.5	4	4.00	13.5	3	6.00	15.3	3												
No. 336	760	5.40	3.05	7.9	3	4.00	12.5	3	6.00	13.5	2												
No. 337	860	5.40	3.05	10.5	3	4.00	16.2	4	6.00	15.8	3												
No. 338	1020	6.70	1.75	7.3	4	2.70	12.7	5	4.70	14.8	3												
No. 345	890	5.70	2.75	12.7	5	3.70	5.1	1															
No. 346	1070	6.90	1.55	7.0	5	2.50	12.6	5															
No. 347	860	6.90	1.55	8.7	6	2.50	8.1	3															
No. 348	980	9.30	...	1.6	...	...	3.1	...															
No. *200[a]	1010	0	1.45	12.0	8	2.40	13.4	6	4.90	14.8	3	5.95	12.9	2	14.45	25.4	2						
No. *202[a]	1640	0	1.45	11.5	8	2.40	10.7	4	4.90	15.2	3	5.95	8.8	2	14.45	19.8	1						
No. *205	1110	0	1.45	9.0	6	2.40	9.5	4	4.90	7.7	2	5.95	5.4	1	14.45	20.3	1						
No. *206	1480	0	1.45	10.0	7	2.40	9.5	4	4.90	12.8	3	5.95	9.5	2	14.45	22.0	2						
No. 215	1210	1.80	...	1.0	...	2.60	2.5	1	4.60	7.0	2	10.70	12.8	1									
No. 216	1630	2.80	...	0.0	...	1.65	3.1	2	3.65	7.4	2	9.70	11.6	1									
No. 217	1080	2.70	...	−6.0	...	1.75	−2.0	...	3.75	4.6	1	9.80	10.2	1									
No. 282	1320	2.80	1.65	1.1	1	3.65	3.0	1	5.65	6.1	1	7.55	13.2	2	11.65	18.9	2						
No. 225	1650	3.90	2.55	4.2	2	4.55	6.1	1	7.50	11.7	2	10.55	13.9	1									
No. 226	1150	3.90	2.55	3.0	1	4.55	5.2	1	7.50	9.8	1	10.55	14.8	1									
No. 227	1640	4.40	2.05	4.6	2	4.05	9.9	2	7.00	12.5	2	10.05	16.2	2									
No. 228	1080	3.80	2.65	0.0	...	4.65	6.0	1	7.60	8.0	1	10.65	12.1	1									

No. 235	1680	5.70	2.75	0.6	...	8.75	8.6	1															
No. 236	1270	6.80	1.65	0.0	...	7.65	4.7	...															
No. 237	1540	4.90	3.55	4.9	1	9.55	16.5	2															
No. 238	1650	4.90	3.55	6.1	2	9.55	13.3	1															
No. 239	1120	4.40	4.05	4.9	1	9.55	12.5	1															
No. 245	1570	5.90	2.55	2.5	1	8.55	11.2	1															
No. 246	1190	6.90	1.55	0.0	...	7.55	10.1	1															
No. 247	1790	5.90	2.55	4.7	2	8.55	16.5	2															
No. 248	1450	6.80	1.65	5.5	3	7.65	7.9	1															
No. 296	1570	6.90	1.55	0.6	...	7.55	9.9	1															
No. 801	1820	8.50	4.00	25.0	6	5.95	27.8	5															
No. *101[a]	2330	0	1.45	8.0	6	2.90	8.0	3	4.90	8.2	2	10.35	15.3	1[b]	14.45	16.6	1						
No. *105[a]	2750	0	1.45	8.2	6	2.90	8.3	3	4.90	8.9	2	10.35	13.4	1	14.45	15.9	1						
No. *102	2110	0	1.45	11.0	8	2.90	8.8	3	4.90	12.9	3	10.35	16.2	1	14.45	15.7	1						
No. *106	2430	0	1.45	4.0	3	2.90	2.9	1	4.90	9.6	2	10.35	10.8	1	14.45	10.6	1						
No. 115	2715	1.70	...	1.3	...	2.70	1.5	1	4.70	4.8	1	10.80	8.5	1									
No. 117	2215	2.70	...	3.5	...	1.75	2.5	1	3.75	5.2	1	9.80	7.5	1									
No. 125	1840	3.70	2.75	2.5	1	5.70	4.6	1	10.75	10.6	1												
No. 127[a]	2540	4.00	2.45	2.0	1	5.40	6.3	1	10.45	10.4	1												
No. 135	2660	4.90	3.55	4.1	1	9.55	10.3	1															
No. 138	1960	4.90	3.55	4.1	1	9.55	8.7	1															
No. 139	1920	4.90	3.55	3.4	1	9.55	8.4	1															
No. 148	2550	6.90	1.55	4.3	3	7.55	6.1	1															
No. 149	1910	6.90	1.55	4.4	3	7.55	7.6	1															
No. 800	2670	9.50	3.00	7.9	3	4.95	10.7	2															

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* Uncoated Wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent copper. All other wires have approximately 0.25 per cent copper.

[b] Summarizes data through 1952

TABLE VIII.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT STATE COLLEGE, PA.

Wire Lot*	Original Breaking Load, lb	Exposure Until First Rust, yr	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test		
			Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss		Exp. Since 1R, yr	Tensile Strength Loss	
				%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr		%	%/yr
No. † 501[a]	500	0	1.3	12.0	9	1.8	11.0	6	2.7	16.0	6	5.3	26.8	5	9.3	33.0	4	14.3	52.0	4
No. † 506	520	0	1.3	17.4	13	1.8	13.5	8	2.7	19.2	7	5.3	27.1	5	9.3	37.5	4	14.3	55.8	4
No. † 507	590	0	1.3	16.0	12	1.8	14.5	8	2.7	16.0	6	5.3	25.7	5	9.3	38.7	4	14.3	56.8	4
No. 510	500	5.7	0.6	1.0	1	1.5	8.0	5	5.6	33.3	6	8.6	32.0	4						
No. 511	280	4.7	1.6	12.3	8	2.5	12.3	5	6.6	38.6	6	9.6	40.4	4						
No. 512	320	4.5	1.8	13.8	8	2.8	12.3	4	6.8	41.5	6	9.8	37.0	4						
No. 520	280	6.3	3.0	−3.6	...	5.0	21.4	4	8.0	14.3	2									
No. 521	270	6.8	2.5	−9.3	...	4.5	22.2	5	7.5	20.3	3									
No. 522	500	7.5	1.8	−4.0	...	3.8	25.0	7	6.8	25.0	4									
No. 530	310	11.3	3.0	20.9	7															
No. 531	500	9.0	5.3	17.7	3															
No. † 400[a]	470	0	1.3	12.5	9	2.3	4.2	2	4.3	11.5	3	9.3	27.1	3	14.3	25.0	2			
No. † 401[a]	440	0	1.3	11.0	8	2.3	3.3	1	4.3	10.0	2	9.3	20.0	2	14.3	32.4	2			
No. † 405	470	0	1.3	13.5	10	2.3	7.2	3	4.3	12.4	3	9.3	21.7	2	14.3	32.0	2			
No. † 406	460	0	1.3	12.8	10	2.3	5.3	2	4.3	11.7	3	9.3	18.1	2	14.3	25.6	2			
No. 410	570	4.5	1.8	...	...	2.8	5.2	2	6.8	6.8	...	9.8	20.2	2						
No. 411	615	5.5	0.8	...	...	1.8	...	...	5.8	18.7	2	8.8	18.2	2						
No. 412	830	5.7	0.6	...	...	1.5	4.2	3	5.6	22.9	2	8.6	18.7	2						
No. 421	470	5.8	3.5	2.1	1	5.5	18.1	3	8.5	16.0	1									
No. 422	500	6.8	2.5	−3.0	...	4.5	16.0	4	7.5	10.0	1									
No. 423	400	5.8	3.5	1.2	...	5.5	20.0	4	8.5	13.8	1									
No. 430	780	8.7	5.6	12.8	2															
No. 431	880	7.7	6.6	21.0	3															
No. 432	510	11.0	3.3	6.9	2															
No. † 300[a]	730	0	1.3	9.5	7	2.3	4.7	2	7.3	13.5	2	14.3	18.9	1						
No. † 302[a]	1100	0	1.3	11.0	8	2.3	12.7	6	7.3	20.0	3	14.3	28.7	2						
No. † 305	1070	0	1.3	9.0	7	2.3	6.9	3	7.3	14.7	2	14.3	22.8	2						
No. † 306	7010	0	1.3	8.5	6	2.3	2.8	1	7.3	12.6	2	14.3	16.9	1						
No. 315	900	3.6	3.3	5.6	2	5.6	3.3	1	10.6	17.1	2									
No. 316	1035	5.7	1.5	...	...	3.6	−5.3	...	8.6	11.1	1									
No. 317	795	6.8	0.5	...	...	2.5	−3.1	...	7.5	10.7	1									
No. 325	730	7.8	6.5	7.5	1															
No. 326[a]	700	6.5	7.8	10.7	1															
No. 327	1160	8.0	6.3	6.8	1															
No. 329	720	6.3	8.0	8.3	1															
No. 335	1040	8.0	6.5	9.6	2															
No. 336	760	10.3	4.0	7.9	2															
No. 337	860	9.7	4.6	11.1	2															
No. 338	1020	12.0	2.3	...	...															

No. † 200[a]	1010	0	1.3	7.3	6	2.3	8.1	4	7.3	15.3	2	14.3	15.8	1					
No. † 202[a]	1640	0	1.3	2.5	2	2.3	3.8	2	7.3	14.0	2	14.3	15.2	1					
No. † 205	1110	0	1.3	9.0	7	2.3	1.8	1	7.3	12.6	2	14.3	9.5	1					
No. † 206	1480	0	1.3	8.8	7	2.3	8.1	4	7.3	12.8	2	14.3	16.9	1					
No. 215	1210	3.6	3.3	5.7	2	10.6	4.6	...											
No 216	1630	5.7	1.5	2.4	2	8.8	3.9	...											
No. 217	1080	5.5	1.8	...	...	8.8	0.9	...											
No 282	1320	6.7	2.6	0.8	...	4.6	8.7	2	7.6	9.8	1								
No. 225	1650	8.0	6.3	7.6	1														
No 226	1150	8.7	5.6	8.3	1														
No. 227	1640	8.7	5.6	8.5	1														
No. 228	1080	7.8	6.5	9.3	1														
No. 235	1680	11.0	3.0	5.3	2														
No. 236	1270	13.0	1.3	−5.5	...														
No. 237	1540	8.7	5.6	37.2	7														
No. 238	1650	9.0	5.3	7.5	1														
No. 239	1120	10.3	4.0	5.1	1														
No. † 101[a]	2330	0	1.3	5.5	4	2.3	3.2	1	7.3	10.3	1	14.3	9.3	1					
No. † 105[a]	2750	0	1.3	6.5	5	2.3	4.7	2	7.3	8.8	1	14.3	5.4	...					
No. † 102	2110	0	1.3	7.5	6	2.3	7.9	3	7.3	0.9	...	14.3	10.3	1					
No. † 106	2430	0	1.3	1.5	1	2.3	2.5	1	7.3	7.7	1	14.3	2.5	...					
No. 115	2715	3.7	3.0	2.3	1	10.6	0.2	...											
No. 117	2215	4.7	2.5	1.1	...	9.6	0.7	...											
No. 125	1840	7.3	7.0	...	...														
No. 127[a]	2540	8.7	5.6	−1.9	...														
No. 135	2660	10.3	4.0	−4.1	...														
No. 138	1960	10.5	3.8	−1.8	...														
No. 139	1920	9.3	4.3	−3.0	..														

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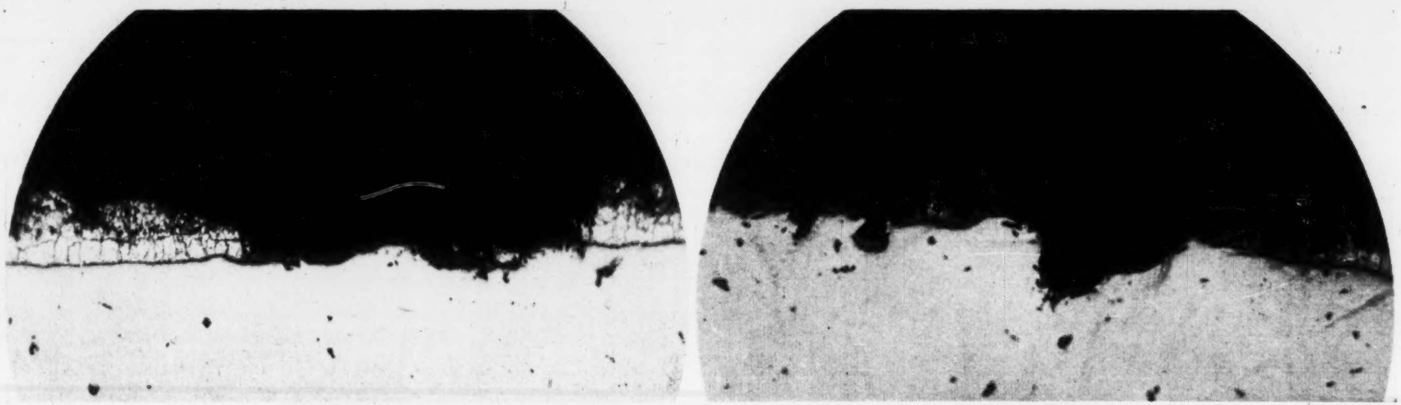
* 500 Series—14½ gage. 400 Series—12½ gage. 300 Series—11 gage. 200 Series—9 gage. 100 Series—6 gage.

[a] Low copper content wires, 0.05 to 0.08 per cent copper. All other wires have about 0.24 per cent copper.

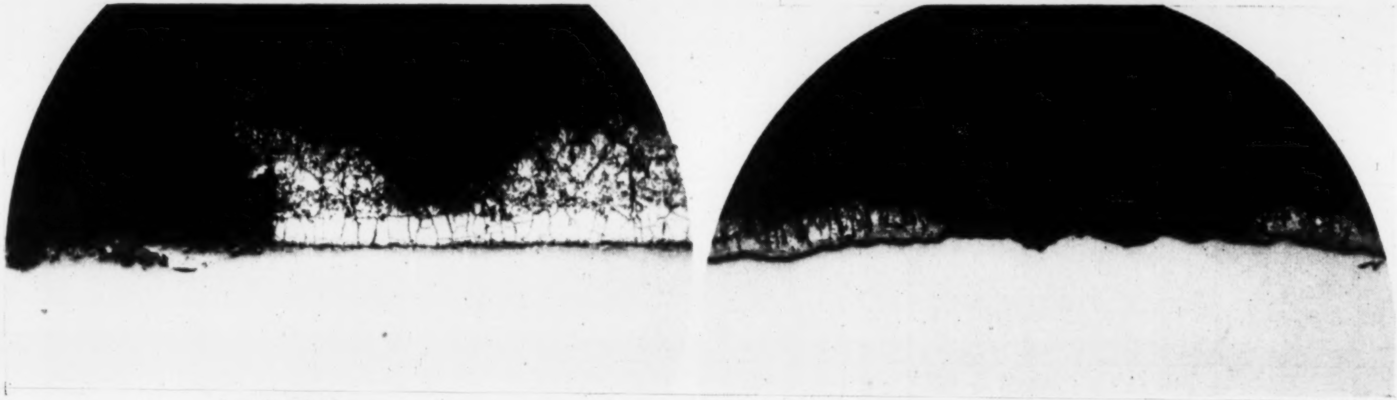
[b] Summarizes data through 1952.

† Uncoated wires. All other wires had zinc coating.



Longitudinal   Transverse

Wire Lot No. 139



Longitudinal
Etchant: 4 per cent Picral   Transverse

Wire Lot No. 247

FIG. 5.—Galvanized Wires Exposed 15 yr at College Station, Tex. Section Through Rough Pitted Area (×250).

REFERENCE INFORMATION

ON UNFABRICATED WIRE, BARBED WIRE AND FENCE IN THE WIRE INSPECTION REPORT.

The Wire Test Committee responsible for planning and arranging the exposure tests of specimens covered in this report recommends that the following statement be made in all reports relating to the samples of wire and wire products under exposure tests:

> "It is emphasized that the weight of coating ranges shown under each weight group have been arbitrarily chosen and purposely held to close limitations by the Committee and do not necessarily individually represent any manufacturers' commercial product. On the other hand, the entire range of coatings covered by all weight groups as a whole are designed to include a sufficient range of coating to furnish the useful corrosion resistance data desired."

SYSTEM OF NUMBERING SPECIMENS

Zinc coated and uncoated steel specimens (all have 3-digit numbers)..................(c)

When the first digit is.......	5	4	3	2	1
the respective gages are:	14.5	12.5	11	9	6

The second and third digits indicate weight of zinc coating as follows:

	oz. per sq.ft.		oz. per sq.ft.
00–04 (LC)*	uncoated	50–59 (CB)	1.6 to 1.8
05–09 (CB)*	uncoated	60–64 (CB)	1.0 to 1.2
10–14 (CB)	0.20 to 0.30	65–69 (CB)	1.3 to 1.5
15–19 (CB)	0.25 to 0.35	70–74 (CB)	1.6 to 1.8
20–24 (CB)	0.35 to 0.45	75–79 (CB)	2.4 to 2.6
25–29 (CB)	0.45 to 0.55	80–84 (CB)	less than 0.50
30–34 (CB)	0.50 to 0.60	85–89 (CB)	1.2 to 1.5
35–39 (CB)	0.60 to 0.70	90–94 (CB)	2.4 to 3.0
40–44 (CB)	0.70 to 0.85	97–99 (CB)	extra heavy
		95–96 (CB)	chromated after coating
45–49 (CB)	0.80 to 1.00 on farm fence and unfabricated wire. 0.85 to 1.00 on barbed wire. 0.80 to 0.95 on wire strand.		

Specimens of Special Materials:
000–015: uncoated corrosion-resistant steel.
020–030: copper-covered steel.
040–050: lead-coated steel.

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* LC = low copper content wire; CB = copper bearing wire

Reference Characters

(a)......Wire lot numbers identify wire from a coil or coils of wire supposedly alike.

Wire Lots of Zinc-Coated or Bare Mild Steel	Usually Appear in the Test as shown by ✓				
	Unfab. + Wire	Int. of + Fence	T and B + of Fence	T and B of Fence	Barbed Wire
100 series	✓ (S)	....	....	....	....
200 series	✓ (S)	✓ (S)	✓ (S)	✓ (300)	....
300 series	✓ (S)	✓ (S)	....	✓ (500)	....
400 series	✓ (S)	✓ (S)	....	....	✓ (400)
(s)...... 500 series	✓ (S)	✓ (S)	....	....	....

(S) The specimen numbers of the unfabricated wire or fence are the same as the wire lot numbers.

(300), (400), (500): Specimens of fence numbered in the series 300 have top and bottom wires of the 200 series wire lots; fences numbered in the 500 series have top and bottom wires of the 300 series wire lots. Specimens of barbed wire (numbered in the 400 series) have wire lots of the 400 series but not necessarily of the same identical wire lot numbers.

Par. A. The zinc-coated wire lots table below shows:

(1) All the individual numbered wire lots and the various utilizations of each wire lot in the test.

(2) The results of weight of coating tests on samples cut from each source (unfabricated wire, fence interiors, fence top and bottom wires, and barbed wires).

(b)...... (3) The average coating for each wire lot which is the average of all the figures in the third and following columns in each row.

These average coating figures are the ones entered in other tables of this report. For example, wherever unfabricated wire sample No. 317 or farm fence sample No. 317 are listed, their indicated weights of coating are 0.38 oz. per sq.ft., and not 0.46 and 0.34, respectively.

These wire lot and coating reference data provide an indication of the uniformity of coating on any wire lot.

Par. B. The makeup of fences table below shows:

(1) The lot number of the wire used in the top and bottom line wires of each fence. (The lot number of the wire used in the interior line and stay wires of any fence is the same as the fence number.)

(2) The type of fence, that is, the combination of gages used in its fabrication as explained below:

(d)...... Type of fence................................	4	3	2	1
Gage of interior line and stay wires....	14.5	12.5	11	9
Gage of top and bottom line wires......	11	10	9	9

+ ......Abbreviations: Unfab., unfabricated wire; Int., interior line and stay wire; T and B, top and bottom line wires.

ZINC-COATED WIRE LOTS: THEIR COATING WEIGHTS AND TEST SPECIMENS IN WHICH THEY ARE USED

MAKEUP OF FENCES

ZINC-COATED WIRE LOTS: THEIR COATING WEIGHTS AND TEST SPECIMENS IN WHICH THEY ARE USED
(See Par. A and reference characters above for explanations) (Coating weights, oz. per sq.ft.)

Wire Lot (a) (Fence)	(b) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(s)	Coating Weight Tests on Samples Cut From Fence Int.(s)	Barbed Wire Coating	Barbed Wire No.
No. 510(c)	0.29	0.29	0.29		
No. 511	0.24	0.24	0.23		
No. 512	0.27	0.27	0.27		
No. 520	0.36	0.34	0.38		
No. 521	0.38	0.40	0.36		
No. 522	0.44	0.44	0.44		
No. 530	0.61	0.59	0.62		
No. 531	0.60	0.58	0.61		
No. 532	0.49	....	0.49		
No. 540	0.70	0.72	0.68		
No. 541	0.76	0.80	0.72		
No. 542	0.70	0.69	0.71		
No. 543	0.79	0.83	0.74		
No. 551	1.69	1.69	....		
No. 552	2.08	2.08	....		
No. 410(c)	0.30	0.25	0.38	0.27	(410)
No. 411	0.28	0.28	0.28	....	....
No. 412	0.29	0.30	0.30	0.27	(412)
No. 421	0.47	0.49	0.49	0.44	(422)
No. 422	0.35	0.38	0.31	....	....
No. 423	0.31	0.35	0.31	0.28	(420)
No. 430	0.54	0.56	0.52	0.53	(431)
No. 431	0.48	0.49	0.48	0.46	(430)
No. 432	0.57	0.58	0.60	0.53	(432)
No. 433	0.49	....	0.49	....	....
No. 440	0.76	0.72	0.79	....	....
No. 441	0.78	0.77	0.76	0.82	(442)
No. 442	0.72	0.65	0.86	0.64	(441)
No. 443	0.74	0.74	0.78	0.70	(443)
No. 444	0.87	0.87	....	....	....
No. 450	1.50	1.48	....	1.52	(452)
No. 451	1.83	1.71	....	1.95	(451)
No. 495 Cr	Made from unfab. wire 443.				

	Unfab. Wire		Unfab. Wire
No. 115(c)	0.25	No. 139	0.64
No. 117	0.27	No. 148	0.90
No. 125	0.41	No. 149	0.85
No. 127	0.54	No. 151	1.86
No. 135	0.74	No. 800(6 ga.)	1.35
No. 138	0.67	No. 801(9 ga.)	1.20

Wire Lot (a) (Fence)	(b) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(s)	Fence Int.(s)	T and B of Fence Top	T and B of Fence Bottom	T and B of Fence No.
No. 315(c)	0.25	0.25	0.25	0.25	0.26	(512)
No. 316	0.32	0.32	0.31	0.32	0.33	(510)
No. 317	0.38	0.46	0.34	0.37	0.36	(511)
No. 325	0.44	0.50	0.52	0.37	0.39	(520)
No. 326	0.38	0.40	0.35	....	....	....
No. 327	0.47	0.48	0.44	0.48	0.48	(522)
No. 329	0.54	0.56	....	0.45	0.61	(521)
No. 335	0.61	0.61	0.63	0.59	0.60	(531)
No. 336	0.66	0.73	0.60	....	....	....
No. 337	0.57	0.66	0.59	0.50	0.52	(532)
No. 338	0.69	0.67	0.76	0.67	0.65	(530)
No. 345	0.84	0.83	0.97	0.76	0.79	(541)
No. 346	0.88	0.84	0.84	0.96	0.88	(542)
No. 347	0.88	0.96	1.00	0.78	0.79	(543)
No. 348	1.05	1.04	1.07	1.05	1.06	(540)
No. 350	1.75	1.73	1.76	....	....	....
No. 351	1.65	1.65	....	....	....	....
No. 352	1.75	1.82	1.68	....	....	....
No. 397	2.80	2.80	2.79	....	....	....

Wire Lot (a) (Fence)	(b) Average Coating	Unfab. Wire(s)	Fence(s) T-B Top	Fence(s) T-B Bottom	T and B of Fence Top	T and B of Fence Bottom	T and B of Fence No.
No. 215(c)	0.28	0.28	0.25	0.25	0.33	0.29	(315)
No. 216	0.28	0.31	0.30	0.31	0.27	0.29	(316)
No. 217	0.36	0.29	....	....	0.40	0.39	(317)
No. 225	0.45	0.45	0.49	0.44	0.43	0.47	(327)
No. 226	0.45	0.46	....	....	0.45	0.45	(326)
No. 227	0.51	0.59	0.48	0.49	....	....	
No. 228	0.50	0.49	0.48	0.48	0.50	0.53	(325)
No. 235	0.67	0.82	0.60	0.62	0.68	0.64	(337)
No. 236	0.68	0.66	0.68	0.73	0.69	0.65	(338)
No. 237	0.63	0.72	0.67	0.55	0.58	0.61	(335)
No. 238	0.60	0.60	0.59	0.61	0.59	0.61	(336)
No. 239	0.65	0.65	....	....	....	....	....
No. 245	0.81	0.80	0.83	0.78	0.81	0.81	(346)
No. 246	0.95	0.97	0.94	0.98	0.89	0.95	(347)
No. 247	0.96	0.94	0.95	1.00	1.01	0.91	(345)
No. 248	0.81	0.66	0.92	0.68	0.91	0.89	(348)
No. 250	1.76	1.76	....	....	....	....	....
No. 251	1.86	1.68	....	....	1.99	1.91	(352)
No. 252	1.69	1.67	1.85	1.57	....	....	(350)
No. 282	0.37	0.37	....	....	....	....	....
No. 296 Cr	Made from unfab. wire 245.						
No. 298	2.72	2.78	2.71	2.74	2.71	2.67	(397)
No. 299	3.00	3.00	....	....	....	....	....

MAKEUP OF FENCES
(See Par. B and reference characters above)

Int. Lot (Fence)	Type (d)	T-B Lot	Int. Lot (Fence)	Type (d)	T-B Lot
ZINC-COATED FENCES					
No. 510	4	No. 316	No. 410	3	....
No. 511	4	No. 317	No. 411	3	....
No. 512	4	No. 315	No. 412	3	....
No. 520	4	No. 325	No. 421	3	....
No. 521	4	No. 329	No. 422	3	....
No. 522	4	No. 327	No. 423	3	....
No. 530	4	No. 338	No. 430	3	....
No. 531	4	No. 335	No. 431	3	....
No. 532	4	No. 337	No. 432	3	....
No 540	4	No. 348	No. 433	3	....
No. 541	4	No. 345	No. 440	3	....
No. 542	4	No. 346	No. 441	3	....
No. 543	4	No. 347	No. 442	3	....
			No. 443	3	....
No. 315	2	No. 215			
No. 316	2	No. 216	No. 215	1	No. 215
No. 317	2	No. 217	No. 216	1	No. 216
No. 325	2	No. 228	No. 225	1	No. 225
No. 326	2	No. 226	No. 227	1	No. 227
No. 327	2	No. 225	No. 228	1	No. 228
No. 335	2	No. 237	No. 235	1	No. 235
No. 336	2	No. 238	No. 236	1	No. 236
No. 337	2	No. 235	No. 237	1	No. 237
No. 338	2	No. 236	No. 238	1	No. 238
No. 345	2	No. 247	No. 245	1	No. 245
No. 346	2	No. 245	No. 246	1	No. 246
No. 347	2	No. 246	No. 247	1	No. 247
No. 348	2	No. 248	No. 248	1	No. 248
No. 350	2	No. 252	No. 252	1	No. 252
No. 352	2	No. 251	No. 298	1	No. 298
No. 397	2	No. 298			
BARE-STEEL FENCES					
No. 505	4	No. 306	No. 305	2	No. 206
No. 506	4	No. 305	No. 306	2	No. 207
No. 405	3	....	No. 205	1	No. 205
No. 406	3	....	No. 206	1	No. 206
Coppered-covered steel fence No. 020			Int. No. 021	2	No. 022
Lead-coated steel fence No. 040			Int. No. 041	1	No. 041

UNCOATED CORROSION-RESISTANT STEEL FENCES
No. 001 (T - B Lot No. 001) Gages: Int. 12 ; T - B 12
No. 002 (T - B Lot No. 002) Gages: Int. 12.5; T - B 12.5
No. 003 (T - B Lot No. —) Gages: Int. 15 ; T - B 13

PLATE II
PROCEEDINGS, AM. SOC. TESTING MATS.
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made of the appearance of each specimen. Prior to the time of rusting the specimens are noted as appearing "metallic," "gray," "yellow," etc. After rusting has begun, estimates are made of the percentage of area rusted on each specimen.

The term "rust" as given in the inspection instruction states: "Rust means real rust of the base metal—ordinarily a rough corroded area." The committee has been in agreement on interpretation of this term in their various inspections, but some of the exposure plots have never been visited by members of the committee.

During the past few years inspection reports from College Station, Tex., and from Santa Cruz, Calif., two of the least corrosive sites, have listed certain galvanized wires as rusting at what appeared to be too early a date for the coating weight and exposure location. Specimens from the 14 wire lots involved (Nos. 541, 440, 444, 345, 247, 430, 431, 336, 337, 235, 238, 239, 138, 139) were removed from the racks after 15-yr exposure, examined by the committee and were all rated GY or Y although the inspection reports had rated them as varying per cent R.

The wires had a speckled, rough appearance, not similar to that observed at locations where corrosion was more rapid. The speckled rough appearance was due to the formation of globules of iron hydroxide on the iron-zinc alloy coating. This seems to be peculiar to exposures in dry warm atmospheres. In atmospheres of rain, snow, or more rapid corrosion, these same coatings weather to dark shiny films.

The specimens were further subjected to stripping tests to determine the weight of remaining coating Chemical analyses of the coatings and photo-micrographs of the corroded areas were made.

The weight of zinc coating remaining as determined by stripping tests varies from 75 to 85 per cent of the original coating weight. The corrosion products on the surface of these wires is mostly a mixture of hydroxide of iron and zinc carbonate plus a considerable amount of chlorides, and some phosphates. There is some excess iron in the coating, unaccounted for except as having come from the base metal. Photomicrographs (Figs. 1 to 5) show that the only areas where the galvanized coating has disappeared are in a few scattered round pits 0.003 to 0.006 in. in diameter. Attack of the steel base metal is, if present, too minute to be recognized in the micrographs.

Findings from Tension Tests:

Tension tests are made on the unfabricated wire specimens. These specimens are exposed in groups of seven comparison samples taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each group when it has lost 5 to 10 per cent of its strength, and the seventh specimen when it has lost some 75 per cent of its strength. There were originally about 840 tensile strength specimens exposed at each site. The number of specimens removed to date is shown in Table B.

Early in 1951, just prior to the abandonment of the Brunot Island (Pittsburgh) test site all the remaining (11) galvanized wires were taken for tensile strength test. The results were reported in the 1951 *Proceedings* together with a summary of the complete tensile strength test data from Pittsburgh.

During the 1951 inspections 57 tensile strength specimens were taken from Sandy Hook, 55 were taken from Bridgeport, and 67 were taken from State College. During the 1952 inspections the only specimens taken were from Sandy Hook where 9 galvanized specimens, 10

TABLE B.—NUMBER OF TENSILE STRENGTH SPECIMENS REMOVED FOR TESTS.

Exposure Site	Number of Wires Removed 1951 and 1952				Total Removed to Date
	Bare Steel	Zinc-Coated	Lead-Coated	Other Types	
Pittsburgh, Pa	0	11	0	0	771
Sandy Hook, N. J	12	54	4	12	462
Bridgeport, Conn	12	43	0	0	392
State College, Pa	19	48	0	0	185
All Other Sites	0	0	0	0	415
Total	43	156	4	12	2225

TABLE C.—TENSILE STRENGTH TESTS OF LEAD-COATED, COPPER-COATED, AND CORROSION-RESISTANT STEEL WIRE EXPOSED 15 YR AT SANDY HOOK, N. J.

Wire Lot	Type	Original		15 yr Exposure Sandy Hook	
		Breaking Load, lb	Elongation, per cent	Breaking Load, lb	Elongation, per cent
No. 001	Cr.D	1230	3.0	1245	2.5
No. 004	Cr.D	2420	3.5	2330	3.0
No. 005	Cr.D	320	2.1	310	2.0
No. 002	Cr.Q	1310	4.2	1340	3.3
No. 006	Cr.Q	2480	4.0	2500	5.0
No. 007	Cr.Q	280	4.7	290	3.7
No. 003	Cr.Ni	1280	0.5	1315	...
No. 008	Cr.Ni	2510	2.3	2525	...
No. 009	Cr.Ni	290	...	310	...
No. 015	Cr.Ni	1250	0.8	1260	0.6
No. 021	Cu Coat	1640	...	1620	...
No. 022	Cu Coat	2320	...	2295	...
No. 040	Pb Coat	1700	9.5	1760	10.0
No. 041	Pb Coat	1070	12.8	1050	7.5
No. 042	Pb Coat	670	13.0	665	7.5
No. 043	Pb Coat	300	17.1	285	...

TABLE D.—LOSS IN TENSILE STRENGTH, PER CENT PER YEAR OF EXPOSURE.

Exposure Site	No. 14½ Gage	No. 12½ Gage	No. 11 Gage	No. 9 Gage	No. 6 Gage
Pittsburgh, Pa	15	10	7	5	3
Sandy Hook, N. J	9	5	4	3	2
Bridgeport, Conn	4	3	2	1	1
State College, Pa	4	2	1	1	...

corrosion-resistant steel specimens, 2 copper-coated specimens, and 4 lead-coated specimens were taken.

The test results from these specimens are included in Tables VI, VII, and VIII which contain all the data accumulated to date for the bare steel and galvanized steel wires at Sandy Hook, Bridgeport, and State College, respectively.

The corrosion-resistant steel, copper-coated, and lead-coated specimens from any location have shown no loss of strength nor change in elongation value to date. The lead-coated wires after 15 yr exposure at Sandy Hook showed no loss of strength, but the coating peeled and flaked off during tensioning, leaving the whole wire bare. The results of the tensile strength tests on these specialty wires after 15 yr of exposure at Sandy Hook are shown in Table C.

On the basis of tests to date the loss in tensile strength by uncoated steel wires and by zinc-coated steel wires after the wire begins to rust is shown in Table D.

This report was prepared by Mr. A. C. Jahn, former chairman of Subcommittee XV.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

F. M. REINHART,

Chairman.

APPENDIX

SOME OBSERVATIONS ON THE PREECE TEST AND STRIPPING TESTS FOR ZINC-COATED WIRES*

By G. A. Ellinger,[1] W. J. Pauli,[1] and T. H. Orem[1]

Synopsis

A report on an investigation of the Preece test published in 1934 by Groesbeck and Walkup concluded that the rates of solution of variously applied galvanized coatings on steel wires depended upon the type of coating and that the test could not be used to determine the coating thickness although it is a satisfactory means for ascertaining the uniformity of coating. Following recommendations of these investigators regarding certain precautions to be taken in conducting the test, a recent evaluation of the Preece test has confirmed the observations concerning the difference in solution rates of different types of galvanized coatings. Electrogalvanized, regular hot-dip galvanized, asbestos-wiped and galvannealed coatings, as well as wires drawn from electrolytic and Prime Western zinc were investigated. Weight losses measured after each Preece dip showed that galvannealed and asbestos-wiped coatings composed largely of zinc-iron alloy dissolved at the slowest rates and that regular galvanized and electrogalvanized coatings consisting largely of free zinc dissolved at a rate approximating that of pure zinc. Anomalous results sometimes encountered in the test were investigated and were found to result from irregularities in the coating or differences in coating components.

The weight of coatings on the various wires was determined by the hydrochloric acid-antimony chloride and the sulfuric acid methods recommended in ASTM Method A 90[2] and in addition by means of a 1:1 hydrochloric acid-water solution. Additional stripping was conducted in accordance with an electrolytic method first recommended by Glazunov and by Britton. All methods produced relatively uniform results. The electrolytic stripping method, in addition to giving results comparable to those obtained by chemical stripping, provided data which, when plotted as time-potential curves, furnished readily interpretable information regarding the processes by which the wire was coated and the relative proportions of alloy layer and pure zinc, where applicable, comprising the coating.

One of the most widely used tests for the inspection of galvanized steel is the Preece test, which consists in placing a sample of the material to be tested in a solution of copper sulfate under standard conditions, and determining the number of 1-min immersions which can be made before bright, adherent copper will plate out on the specimen due to the exposal of the underlying steel. The method was first used by Pettenkofer (1)[3] in testing galvanized telegraph wires for the Bavarian Railways in 1848, and, about 1880, was also used by Sir William Henry Preece (from whom the test took its name) for the same purpose. The test has been included in many specifications for galvanized materials and, practically ever since its inception, has been the subject of considerable criticism. Although intended primarily as a test to determine areas of minimum thickness of coating, the results have frequently been distorted as indicating total weights of coating and useful life of the product in service. Patrick and Walker (2) in 1911 and Bablick (3) in 1926, among others, reveal that there was a very marked difference of opinion as to the real significance of the test. In 1934, Groesbeck and Walkup (4) published the results of a comprehensive investigation of the Preece test including a study of the composition of the solution, preparation of the specimen, end points, and thickness distribution of the coatings. The present ASTM Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 - 41)[4], which is widely used, had its foundation in the work of Groesbeck and Walkup.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Chief, Chemist, and Metallurgist, respectively, Corrosion Section, National Bureau of Standards, Washington, D. C.

[2] Standard Methods of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A 90 – 39), 1952 Book of ASTM Standards, Part 1, p. 1015.

There are many recognized inconsistencies in the results of the test. Certain types of coatings are reported to withstand more "Preece dips" than the same thickness of other types; for example, pure zinc coatings applied electrolytically are said to withstand fewer dips than other coatings. There also appeared to be a lack of consistency among results even from the same type of coating. Due to these inconsistencies there has been an increasing demand in recent years for the removal of this test from wire specifications as ASTM Committee A-5 on Corrosion of Iron and Steel did to sheet and plate as long ago as 1911. Consequently, at the request of Subcommittee VII on Methods of Testing of Committee A-5, the National Bureau of Standards undertook an investigation of some of the features of the test, as reported herein.

TABLE I.—TYPE AND WEIGHT OF GALVANIZED COATINGS.

Sample	Type of Coating	Stripping Methods, oz per sq ft				Other Methods, oz per sq ft	
		HCl conc	HCl (1:1)	H_2SO_4, 4 per cent	Electrolytic	Magnegage	Metallographic
A	Electrogalvanized	0.97	0.96	0.97	0.96	0.98	0.94
B		0.51	0.51	0.51	0.48	0.54	0.26
C	Regular Galvanized	1.00	0.98	1.00	0.95	1.04	0.79
D		0.92	0.91	0.96	0.90	0.90	0.84
E		1.25	1.24	1.24	1.20	1.22	1.11
F		1.20	1.17	1.18	1.18	1.34	1.00
G	Asbestos Wiped	0.51	0.52	0.52	0.50	0.54	0.44
H		0.42	0.40	0.39	0.41	0.43	0.46
J		0.27	0.26	0.27	0.24	0.25	0.23
K	Galvannealed	1.35	1.38	1.38	1.22	1.55	1.37
L		0.68	0.66	0.68	0.64	0.75	0.66
M		0.72	0.72	0.74	0.67	0.89	0.72
N		0.25	0.26	0.26	0.22	0.26	0.18

MATERIALS

Thirteen samples of wires representing four types of coatings, with two or more thicknesses of zinc for each type are shown in Table I. Originally it was planned to investigate four different weights of coatings for each type so that direct comparisons of types and weights could be made. At the time the testing was started, not all of these were obtained, however, and thus could not be included. Consequently, there is not a complete series-of-four coating weights for any of the types. Heavily coated unwiped wires submitted by the manufacturers as Double Galvanized, Type 3, etc. are designated as regular galvanized in the table, while those which have been run through asbestos pads (Single Galvanized, Tight Wiped, etc.) are designated asbestos wiped. Galvannealed refers to those coatings which have been applied by hot dipping and subsequently heat treated. As a control and for comparison purposes electrolytic zinc rods swaged to 0.195 in. in diameter were used. Similar rods swaged from Prime Western spelter were also used, but the results of the tests were so similar to those of the electrolytic zinc that they are not included here.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 135.

[4] 1952 Book of ASTM Standards, Part 1, p. 1066.

EXPERIMENTS

Weight of Coating:

Preliminary to the Preece test determinations the weights of the coatings on the various wires were obtained by three conventional methods of acid stripping, by electrolytic stripping, by Magne-gage measurements, and by metallographic examination. The standard concentrated hydrochloric acid-antimony chloride solution described in ASTM Standard Method A 90[2] was used as a control, and the coating weights obtained by this method are used throughout this report except where noted. A dilute hydrochloric acid solution, made by mixing equal parts of concentrated hydrochloric acid and distilled water, has been proposed as an alternate for the standard ASTM solution. The 4 per cent sulfuric acid solution[2] used as an alternate method in determining the weight of coating on galvanized sheets, but not specified for wire, was also used for comparison with the standard method.

Electrolytic stripping was conducted in accordance with the method independently developed and contemporarily used by Glazunov (5) and by Britton (6). A cell similar to that used by Britton was employed, in which the specimen was made the anode and a piece of perforated zinc sheet the cathode. The electrolyte consisted of 20 parts of cp sodium chloride and 10 parts of cp hydrated zinc sulfate dissolved in 100 parts of distilled water. The specimen was placed in the center of a 600-ml beaker with the cathode curved around the inside wall. The temperature was controlled by immersing the beaker in a water bath which was maintained at 25 ± 2 C. The current density maintained on each specimen (about 300 ma per sq in.) was determined experimentally as that which would remove the zinc coating at a slow enough rate to permit satisfactory readings and to prevent heating of the solution. Readings of voltage and amperage were made every 30 sec and the weight of the coating removed was determined from the number of coulombs used, assuming a 100 per cent anode efficiency calculated on the basis of pure zinc.

Magne-gage measurements of the coating thicknesses were made peripherally at about 25 places along a 6-in. length of each wire.

Microscopic measurements of the coating thicknesses were also made on cross-sections of the wires which had been copper plated for protection of the edges and then mounted in Bakelite and polished in accordance with a method described by Rowland and Romig (7). The alumina-water mixture used in the polishing operation was maintained at a pH of 7.0 to 7.6 in order to prevent pitting of the coatings. The polished specimens were etched in a solution of 3 drops of concentrated HNO_3 in 50 ml of amyl alcohol.

Results of the measurements of the total weight of coating for each of the materials by all of these procedures are given in Table I. There were no significant differences in the results of stripping by the three acid methods. Electrolytic stripping produced results similar to those of the acid methods but tended to indicate slightly lower weights of coatings, particularly in the galvannealed samples. Magne-gage readings were generally in good agreement with the stripping results except for the galvannealed materials and specimen F (regular galvanized) where appreciably higher values were indicated by the Magne-gage readings. The metallographic observations of coating thicknesses generally indicated lower values than those obtained by other methods. This method, of course, is subject to variations in polishing and etching techniques, and is not generally used for determining the thickness of galvanized coatings.

During electrolytic stripping each type of coating produced a distinctly characteristic potential-time curve, Fig. 1. The original curves were drawn from points plotted at $\frac{1}{2}$-min intervals. For electrolytic zinc the cell potential was about 290 mv and for the steel basis metal about 850 mv. The total thickness of a specific type of coating is proportional to the length of time required for stripping to basis metal, provided that the current is maintained at the same

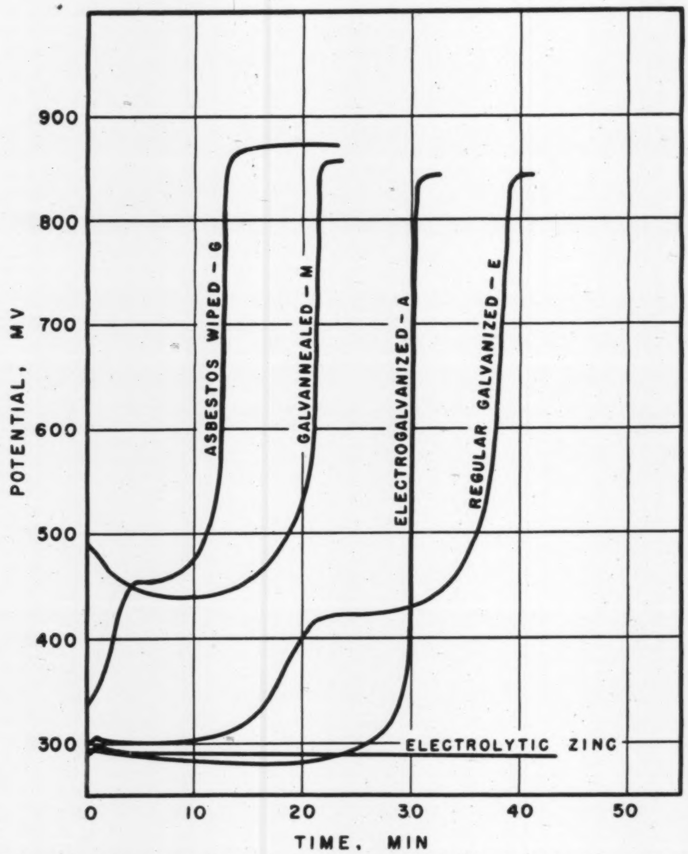

FIG. 1.—Representative Potential-Time Curves for Electrolytic Stripping of Various Types of Coatings and Electrolytic Zinc.

value. For example, electrogalvanized wire *A*, containing approximately a 1-oz coating was completely stripped and reached the steel potential after about 30 min, while electrogalvanized wire *B*, containing about half as much coating reached the steel potential after about 15 min. Barring eccentricity for a given type of coating, the only influence of the weight was to displace the curve for a thinner coat to the left in the direction of shorter stripping time; the characteristic shape of the curve for that coating was not affected. Typical curves for electrolytic zinc and the four types of coatings are shown in Fig. 1; different coating thicknesses were deliberately selected for the various types of coating to avoid confusion in the chart and to illustrate clearly the different features of the curves.

For electrolytic zinc and for coatings containing an outer layer of pure zinc represented by specimens A and E, the potential of the cells increased during the first minute after which they gradually decreased to values which remained constant for several minutes. In the case of the electrolytic zinc specimen, as might be expected, the potential, was constant at about 290 mv for the duration of the test, more than 40 min. The electrogalvanized wires which most nearly exhibit the ideal condition of zinc on iron with no intermediate phases, represented by material A, likewise assumed a constant potential which held for about 23 min, and after 30 min sharply rose to the value for steel. The fact that there is a curve between 23 and 30 min may be taken as indicative of a slight nonuniformity in the coating or a slight nonuniformity in current distribution. There were no intermediate breaks in the curve. The regular hot-dipped galvanized wires, represented by material E, showed an intermediate plateau, the potential leveling off at about 420 mv and then increasing rapidly with time to the steel potential of about 850 mv. The intermediate leveling is due to the presence of a zinc-iron alloy which has a cell potential somewhat greater than that of zinc but considerably less than that of steel.

Judging from the shape of the curve for the asbestos-wiped wires, represented by *G*, the knowledge obtained from curves *A* and *E* indicates that this type of coating does not contain an outer free zinc layer, but rather a thin layer of mixed zinc and zinc-iron alloy and a second layer of zinc-iron alloy. This type of curve is similar to that which would be obtained if, in the wiping process, the first 16 min had been removed from curve *E*.

The galvannealed wires represented by *M*, Fig. 1, have an initial potential considerably higher than that of pure zinc and somewhat greater than the level for zinc-iron alloys as found in asbestos-wiped wires. This high initial value observed on all galvannealed specimens, regardless of thickness of coating, indicates a surface layer of different composition, perhaps an oxidized layer. The potential drops from this value to about that of the zinc-iron alloys in the other curves and then rises rapidly to the steel potential.

While the acid-stripping methods used furnish information only on the total thickness of coating, the electrolytic method makes it possible to determine from the curve the type of coating, its total thickness, and the number and approximate thickness of constituent layers. While the stripping times used in these experiments were long, about 40 min for the heaviest coatings, these can be reduced to 5 min or less by the use of higher currents. However, increasing the stripping rate will provide less information concerning the constituents of the coatings themselves. The method appears to be capable of providing much information regarding these coatings.

Preece Test:

Preece tests were made on samples of all of the materials in accordance with the recommendations of Groesbeck and Walkup (4) and the ASTM Standard Method of Test A 239 – 41.[4] The test samples were 6 in. long with 1 in. of each end coated with an acid-resisting enamel so that 4 in. of the galvanized wire were completely immersed in the Preece test solution. According to the standard procedure, the specimens were given dips of 1 min each to failure, the end point being the last dip prior to the appearance of an adherent copper coating which deposited from the solution as a result of baring of the steel. When the test program was being developed it was suggested that ½-min Preece dips also be included. Dips of this duration are included in some specifications for wires containing light weight coatings. However, there was some question of the value of ½-min dips inasmuch as some belief existed that the dip time was misleading and that two ½-min dips did not equal one 1-min dip. The number of dips on the various materials is given in Table II.

TABLE II.—PREECE TEST—NUMBER OF DIPS AND WEIGHTS OF COATINGS.

Sample	Type of Coating	Number of Dips		Weight of Coating, oz per sq ft			Coating Removed, per cent[c]
		1 min	½ min	by HCl (conc) Total	by Preece Test[a]		
					At failure[b]	Avg. per 1-min dip	
A	Electro-galvanized	5	8	0.97	0.92	0.15	95
B		3	5	0.51	0.50	0.13	98
C	Regular Galvanized	5	8	1.00	0.79	0.13	79
D		7	9	0.92	0.89	0.11	97
E		8	12	1.25	1.18	0.13	94
F		10	16	1.20	1.08	0.10	90
G	Asbestos Wiped	3	6	0.51	0.43	0.11	84
H		3	5	0.42	0.33	0.08	79
J		2	4	0.27	0.23	0.08	85
K	Galvannealed	12	17	1.35	1.10	0.09	81
L		10	16	0.68	0.62	0.06	91
M		6	10	0.72	0.56	0.08	78
N		2	5	0.25	0.21	0.07	84
Electrolytic Zinc Wire		...	...	...	..	0.16	...

[a] 1-min dips.

[b] Dip on which copper was deposited; one more dip than in column 3.

[c] Expressed as:

$$\frac{\text{Coating removed in Preece Test (at failure)} \times 100}{\text{Coating removed in Stripping Test (HCl)}}$$

No difficulty was encountered with false end points (premature copper deposits) except for material L (galvannealed). Premature copper deposits occurred on these wires after the first few dips, but these could always be removed by vigorous brushing. These deposits were of the dark color typical of premature end points, and not of the bright color of the true deposit.

To determine the amount of coating removed during each dip, specimens, prepared as previously described, were weighed at the start of the test and after each dip, the loss of coating weight being calculated in ounces of zinc per square foot of surface area. A fresh solution was used for each dip and the zinc and iron contents of the discarded solution were determined. In general the iron contents were not large enough to justify corrections in the loss of weight. The weights of coatings removed at the end of the Preece test (1-min dips) and the average weight removed during each dip are given in Table II. The loss in weight is that determined for the dip on which copper deposited, this being one more than that given in column 3 of the table.

The weight of coating, dissolved in successive dips, either 1 or ½ min, was practically constant except for a slight change in rate of the regular galvanized wires when the outer free zinc coating was removed and the zinc-iron alloy bared. The number of ½-min dips, however, was only about 60 per cent greater than the number of 1-min immersions, indicating that more than half of the total coating removed in 1 min was removed in the first half minute. This relationship held for all types of coatings.

It will be noted that not all of the zinc coating is removed in the Preece Test, the amount ranging from 78 to 98 per cent of that removed by stripping with the concentrated HCl solution. At least 95 per cent of the coating was removed from the electrogalvanized wires and, with one exception, 90 per cent or more from the regular galvanized wires. The coatings on these wires consisted either entirely or partly of free zinc. On the other hand, coatings consisting largely or entirely of zinc-iron alloy were only 85 per cent or less removed except for one galvannealed wire from which 91 per cent was removed.

The results show that the electrogalvanized samples had very uniformly distributed coatings and that when the end point was reached in the Preece Test, the wires were nearly completely covered with copper. As additional evidence of the nature of the coating, the average weight of coating removed for each 1-min dip approached that of pure electrolytic zinc (0.16 oz per sq ft). This is further verified by the microscopic examination which revealed that the coating on each wire was approximately the same thickness around its circumference and of uniform composition.

For regular galvanized wire there is no good relationship between the number of Preece dips and the total weight of coating. Sample D with a coating of 0.92 oz per sq ft withstood 7 Preece dips, whereas sample C with a 1.00 oz coating withstood only 5 dips. However, only 79 per cent of the coating on sample C was removed at failure, while 97 per cent of that on sample D was removed. Examination of the samples after failure indicated that the coating on sample D had been almost completely removed and that the copper had deposited uniformly on the wire, while that on sample C was very eccentric, being estimated to be approximately twice as thick on one surface as on that diametrically opposite. Microexaminations of cross-sections of wires from the same lot confirmed these visual observations. Obviously, the coating of sample C was removed entirely only along a line on one surface at failure, approximately 20 per cent remaining on the wire. Similar inconsistencies in the number of Preece dips and the total weights of coatings are also noted for specimens E and F. These coatings, however, were quite uniform as evidenced by the percentage removed and by microexamination. The possible reason for these differences is discussed later. The average weight of coating removed from the regular galvanized wires for each dip was slightly less than that for electrogalvanized wires.

The asbestos-wiped wires had light coatings 0.5 oz per sq ft or less. The copper deposits at failure were generally local and nonuniform with considerable coating remaining on the wires. On sample H the failure was along a single line and microscopic examination revealed some eccentricity of coating. The average weight removed per dip was considerably less than that for the electrogalvanized wires.

The heaviest and the lightest coatings investigated were found on galvannealed wires. Similar to the regular galvanized samples there are some inconsistencies in the number of dips and the total weight of coating, for example, samples L and M had approximately the same weights of coating but differed widely in the number of dips at failure. Sample M which withstood 6 dips had only 78 per cent of its coating removed, while sample L which withstood 10 dips had 91 per cent of its coating removed. Failure of specimen M was along a single line and most of the surface of the wire still retained its coating. The copper deposit on specimen L was considerably greater than on specimen M and while not perfectly uniform was more evenly distributed. The eccentricity of the coating of specimen M was confirmed by microscopic examination. The copper deposits on the other two specimens K and N were considerably more spotty than that on sample L. The average weight of coating removed for each dip was approximately 0.08 oz per sq ft.

It should be pointed out at this time that the average amount of coating removed for each dip apparently reflects the type of coating on the sample. Pure zinc is dissolved in the Preece Test solution at the average rate of about 0.16 oz per sq ft per min. Electrogalvanized coatings, containing pure zinc only are removed at the average rate of about 0.14 oz per sq ft per min, while regular galvanized coatings consisting partly of free zinc and partly of zinc-iron alloy are removed at an average rate of about 0.12 oz per sq ft per min. Asbestos-wiped coatings containing small amounts of free zinc but mostly zinc-iron alloy and galvannealed coatings consisting entirely

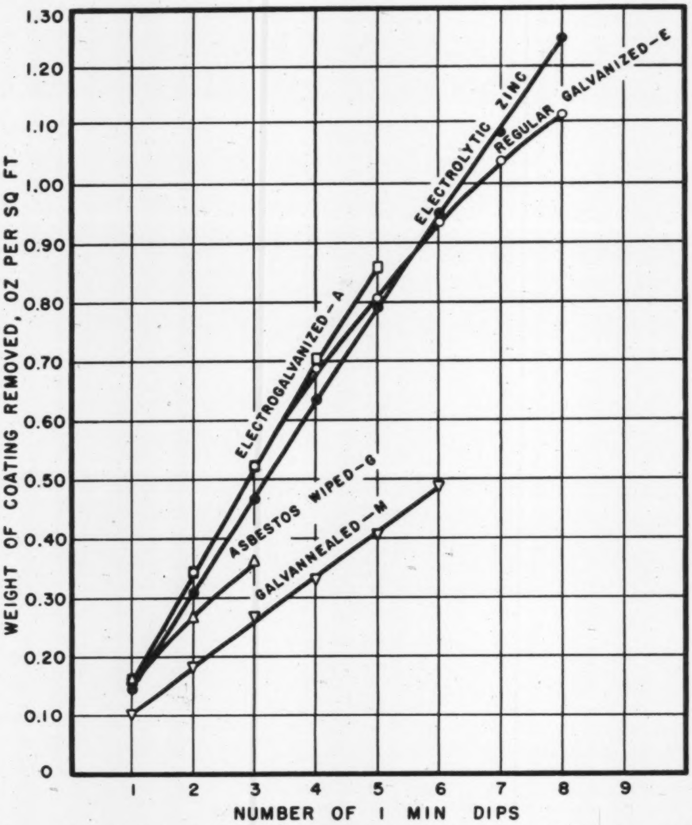

FIG. 2.—Comparison of Cumulative Weight Losses of Various Types of Coatings and Zinc During 1-min Preece Dips.

of zinc-iron alloy have removal rates of about 0.08 oz per sq ft per min, about half of that of pure zinc.

All of the curves for a given type of coating were similar, except that the total weight of coating affected the number of dips to failure. Representative curves for each type of coating are shown in Fig. 2 for 1-min dips and in Fig. 3 for ½-min dips. These curves are for the same samples as in Fig. 1. Curves from specimens of electrolytic zinc wire are also shown for comparison in each figure.

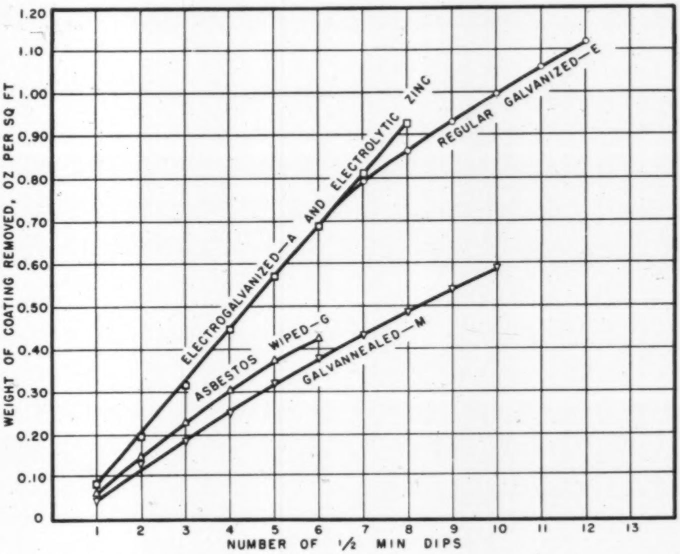

FIG. 3.—Comparison of Cumulative Weight Losses of Various Types of Coatings and Zinc During ½-min Preece Dips.

The curves of Figs. 2 and 3 indicate that in the early stages of the test the rates of solution of the electrolytic zinc, the electrogalvanized wires, and the regular hot-dip galvanized wires are similar. These coatings are composed either entirely of zinc or contain an outer zinc layer (regular galvanized) and solution would be expected to be at approximately the same rates. After the zinc layer is removed from the regular galvanized wires the rate changes, reflecting the exposure of the zinc-iron alloy layer.

Coatings of asbestos-wiped wires composed largely of zinc-iron alloy but containing small amounts of free zinc have considerably lower solution rates as indicated by the smaller slopes of the curves. The slope of these curves is about the same as for the galvannealed wires, which are composed entirely of zinc-iron alloys.

The solution rates of the various coatings indicate the reason for the difference in number of dips which these coatings will withstand. For example, the slope of the curve for removal of the galvannealed coating, Fig. 2, is approximately half of that for the coatings composed entirely or largely of pure zinc. This indicates that the coatings which are composed almost entirely of zinc-iron alloy should dissolve at about half the rate of the pure zinc coatings and should withstand approximately twice the number of dips. The number of dips that a uniformly coated regular galvanized wire should withstand would depend not only upon the total thickness of the coating, but also upon the relative amounts of pure zinc and zinc-iron alloy. Materials E and F furnish examples of this observation. Both have coatings approximately 1.2 oz per sq ft. Material E, however, withstood 8 dips while material F withstood 10. Examination of the microstructures indicate that the coatings are uniformly distributed, but that of material E contains considerably more free zinc than that of material F. This is further shown in the results of electrolytic stripping experiments which indicate that the coating of material E contains about 40 per cent of pure zinc layer and that of material F contains about 30 per cent.

The foregoing indicates that the number of dips that a galvanized wire will withstand varies with type, uniformity, and weight of the coating. As Groesbeck and Walkup pointed out, the Preece test should not be used as a test for weight of zinc coating or for an indication of performance in service until the relative merits of zinc and zinc-iron alloys can be established by service exposure tests.

CONCLUSIONS

The number of dips which a galvanized coating will withstand in the Preece test is dependent not only upon its thickness but also upon its uniformity, composition, and type. Zinc-iron alloys dissolve in the standard solution at about half the rate for pure zinc. Consequently, electrogalvanized coatings, consisting entirely of pure zinc, fail after only half as many dips as comparable weights of galvannealed coatings, which consist entirely of zinc-iron alloy. The number of dips obtained on regular hot-dip coatings depends upon the relative amounts of pure zinc and zinc-iron alloy; coatings consisting largely of pure zinc with only a small amount of alloy will withstand fewer dips than those composed of a thin outer layer of zinc and a large amount of alloy. The Preece test therefore cannot be used as a measure of the thickness of the zinc coating.

The Preece test is capable of providing information concerning the uniformity and distribution of the zinc and whether or not there are thin spots in the coating. Where such information might be used, as in irregular shaped articles or in parts formed or deep drawn from zinc-plated steel, the test could be of some value.

False end points were not a serious problem in this investigation, although they did occur on one galvannealed coating. Proper cleaning precautions minimized the difficulty.

Advantage was taken of the various types and thicknesses of coatings supplied to investigate several methods of stripping to determine the weights of coatings. The results of stripping with concentrated hydrochloric acid, dilute hydrochloric acid (1–1) in water, and 4 per cent sulfuric acid in water were very similar.

An electrolytic stripping method using a sodium chloride-zinc sulfate electrolyte was used to determine the nature of the various coatings. Each type of coating produced a characteristic curve which indicated both the total thickness of the coating and the nature and thickness of each layer. The electrolytic stripping procedure yields more information than is obtained from acid stripping.

Acknowledgment:

Special acknowledgment is made to W. E. Buck, former chairman of Subcommittee VII of Committee A-5, for his interest in the project and valuable assistance in the procurement of samples. The authors also wish to express their thanks to the following companies for providing materials for the investigation: American Steel and Wire Co., Bethlehem Steel Co., Continental Steel Corp., Indiana Steel and Wire Co., Jones and Laughlin Steel Corp., Page Steel and Wire Division of the American Chain and Cable Co., Inc., Keystone Steel and Wire Co., and Republic Steel Corp.

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REPORT OF COMMITTEE A-6

ON

MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties held two meetings during the year: the first in New York, N. Y. on June 23, 1952, during the Annual Meeting, and the second at Niles, Ohio, on March 2, 1953.

The changes in personnel of the committee during the year include the appointment of W. T. Sackett, Jr., to represent Battelle Memorial Inst., and P. P. Cioffi to succeed V. E. Legg as representative o the Bell Telephone Laboratories. The committee regrets the loss of R. C. Taylor as its secretary resulting from the withdrawal of the Western Union Co. from membership on the committee. Milan Getting, Jr. has been appointed to fill the unexpired term as Secretary. R. G. Fernald replaces A. E. Swickard as representative of the Western Electric Co., and D. I. Gordon replaces D. S. Muzzey, Jr. as representative of the Naval Ordnance Laboratory.

The committee has four active task groups working on (*a*) specifications for electrical sheet and strips, (*b*) normal variability of magnetic properties of electrical sheet, (*c*) a manual of magnetic testing to supplement the standard testing specifications, and (*d*) a thoroughgoing revision of the specifications for a-c testing. These groups are all active and are making progress.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of the following standards[1] be approved for adoption as standard:

Methods of:

Testing Magnetic Materials (A 34 – 49), and

Test for Permeability of Feebly Magnetic Materials (A 342 – 49).

The recommendations appearing in this report have been submitted to letter ballot of the committee; 27 members returned their ballots. Following is a summary of the results:

	Affirmative	Negative	Not Voting
Testing Magnetic Materials	27	0	4
Test for Permeability of Feebly Magnetic Materials	26	0	1
No Returns—5			

This report has been submitted to letter ballot of the committee, which consists of 32 members; 27 members returned their ballots, of whom 26 have voted affirmatively and 0 voted negatively.

R. L. SANFORD,
Chairman.

M. GETTING, JR.,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS*

Committee A-7 on Malleable Iron Castings held two meetings during the past year: the first at Atlantic City, N. J., on June 24, 1952 and the second in Detroit, Mich., on March 3, 1953.

During the year, the committee lost the valued services of George E. Bean, whose long-time and active interest in malleable iron developments put him in position to contribute much to the work of Committee A-7 and to the furtherance of ASTM interests. George J. Behrendt of the Naugatuck Works of the Eastern Malleable Iron Co. will serve as Mr. Bean's successor in representing his organization on the committee.

F. T. McGuire has been substituted as the Committee A-7 representative of Deere & Company since the retirement of Hyman Bornstein, but the committee is fortunate in having the continued interest and active help of Mr. Bornstein through his representation, on the committee, of the American Foundrymen's Society. E. B. Fields will represent the Santa Fe Railroad since E. E. Chapman's retirement, and, for like reason, E. L. Layland will represent the Westinghouse Company in place of C. T. Eakin.

At the present time the committee consists of 55 members, of whom one is classified as "not voting"; of the 54 voting members, 27 are classified as producers, 14 as consumers, and 13 as general interest members.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts for Railroad, Marine, and Other Heavy Duty Service (A 338 - 51 T)[1] be continued as tentative without revision.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Chemical and Physical Methods of Testing (L. G. Toye, chairman) has been giving consideration to impact testing of the various types of malleable iron through the use of 0.505-in. diameter cantilever type specimens. Work on this project is being continued with interest centered on normal, subnormal, and elevated temperatures.

This report has been submitted to letter ballot of the committee, which consists of 55 members, one classified as "not voting"; 41 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

JAMES H. LANSING,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-9

ON

FERRO-ALLOYS*

Committee A-9 on Ferro-Alloys held its last meeting in New York City on June 24, 1952. The principal item of business at this meeting was consideration of a proposed change in the wording of the scope of the committee. This committee then recommended to the Joint Coordinating Committee of Committees A-1, A-9, A-10, B-2, and B-4 the following wording:

> "The formulation of specifications covering additive materials such as metals not commercially refined, ferro-alloys, and other metals and metal compounds used in melting operations in the steel and associated metal industries."

This revision was presented to the members of Committee A-9 and the resulting ballot showed 17 affirmative votes, no negative votes, with 1 marked "not voting."

The Joint Coordinating Committee reported on October 31, 1952, on its letter ballot of August 19. This canvass showed 7 affirmative votes, 1 "not voting," and no negative votes.

The new scope was made official when the Board of Directors of the American Society for Testing Materials approved the revision.

With the formal adoption of the revised scope, a committee appointed on June 29, 1949, to prepare By-laws for Committee A-9, was able to function. By-laws were prepared, distributed to the membership, and adopted unanimously.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Specifications for Ferromanganese (**A 99 – 50**):[1]

Subcommittee I on Specifications has received criticisms from the American Iron and Steel Institute and the United States Steel Co. regarding Specification A 99 for Ferromanganese. Their contention is that it is extremely difficult to meet the requirement for 78 to 82 per cent manganese in the standard (7.5 per cent max carbon) grade considering the character of the manganese ore largely available now for the manufacture of this alloy. The committee has prepared a revision of this specification which was referred to the members of Committee A-9 for letter ballot. This proposed revision presents two grades of the standard ferromanganese instead of one, grade A being the same as now specified, and grade B containing 74.0 to 78.0 per cent manganese with impurities the same as for grade A. The vote on this revision was 25 affirmative, 0 negative, and 3 not voting.

This report has been submitted to letter ballot of the committee, which consists of 28 members; 25 members returned their ballots, of whom 24 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. BOWDEN, JR.,
Chairman.

G. F. COMSTOCK,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held meetings at New York, N. Y., on June 26, 1952, during the Annual Meeting of the Society, and on March 3, 1953, at Detroit, Mich.

The present committee consists of 97 members, of whom 41 are classified as producers, 41 as consumers, and 15 as general interest members.

W. F. Hodges and R. H. Heyer have been designated to represent Committee A-10 on the Committee E-1 Task Groups on Methods of Hydrostatic Pressure Testing and Methods of Bend Testing, respectively.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Iron-Chromium and Iron-Chromium-Nickel Alloy Tubular Centrifugal Castings for General Applications (A 362 - 52 T).

Revision of Tentative Recommended Practice for:

Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 44 T).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the new and revised tentative specifications appear in the 1952 Book of ASTM Standards, Part 1.

NEW TENTATIVE

The committee recommends that the proposed Tentative Specification for Stainless Steel Wire Strand be accepted for publication as tentative, as appended hereto.[1]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee A-10 joins with Committee A-1 on Steel in recommending for immediate adoption revision of the following standards and accordingly asks for a nine-tenth affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 - 47**).[2]

Section 11(a).—Revise to read as follows:

> From each group of 100 finished tubes or fraction thereof, one tube shall be selected at random for the tension test specified in Section 6.

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 - 47**).[2]

Table II.—Change the weight tolerances from the present "3.5 per cent over and 5 per cent under" to read "5 per cent over and 5 per cent under."

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 1.

[2] 1952 Book of ASTM Standards, Part 1.

Section 12(a).—Adopt as standard the tentative revision issued October 20, 1951, and published as an editorial note to the specifications. This tentative revision covers the reduction of number of tension tests required.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman).—The principal activities of this subcommittee during the past year included the following:

Specimens to compare the behavior of straight chromium and chromium-nickel stainless steel sheets as used in architectural applications in the Pittsburgh atmosphere were prepared and exposed by a subgroup under the chairmanship of C. P. Larrabee of the U. S. Steel Corp. The assistance of the Stainless Steel Technical Committee of the American Iron & Steel Inst. was secured in providing the large number of test specimens that will be required for the full scale program of atmospheric tests that has been under consideration for several years. Final details of specifications for test pieces that have been allocated to the several producers are being worked out by subgroups. It is hoped that specimens can be exposed before the end of 1953.

A subgroup under the chairmanship of M. H. Brown of duPont has been engaged in preparing a footnote for the Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 44 T), with particular reference to its use for certain grades of stainless steel, particularly Types 321, 316L, and columbium-modified 316. It is expected that this will be presented for action during the June 1953 meeting of the committee.

Attention is being given the possible adoption of a newly developed electrolytic oxalic acid etching technique as a means for screening stainless steels from the boiling nitric acid test, Recommended Practice A 262. This technique was described in a paper by M. A. Streicher entitled "Screening Stainless Steels from the Nitric Acid Test by Electrolytic Etching in Oxalic Acid,"[4] which was sponsored by the subcommittee. Subsequently, with the cooperation of M. H. Brown and M. A. Streicher of the E. I. duPont de Nemours Engineering Experiment Station, specimens of different grades of stainless steel in different conditions of heat treatment were distributed among 17 cooperating laboratories in a round-robin test program to compare the etching technique with the results of the standard boiling nitric acid test. The results of this effort are being summarized by a subgroup under the chairmanship of M. H. Brown.[5] Consideration will be given to adding this technique as a supplement to Recommended Practice A 262.

In accordance with action taken by Subcommittee IV and by Committee A-10, broken Charpy impact test specimens from the studies of sigma phase that were undertaken by Subcommittee VI were turned over to Subcommittee IV for tests in boiling nitric acid as per Recommended Practice A 262. These tests were carried out by M. H. Brown at the duPont Engineering Experiment Station and the results were turned over to Subcommittee VI. Arrangements are being made for metallographic study of thes pecimens from these acid tests, to be followed by a joint subcommittee report.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] Published in ASTM BULLETIN, No. 188, February, 1953, p. 35 (TP27).

[5] The papers giving the results of this test program appear in ASTM BULLETIN, No. 195, January, 1953: F. H. Beck, N. D. Greene, Jr., and M. G. Fontana, "Electrolytic Etching in Oxalic Acid Used to Screen Cast CF-8 and CF-8M Stainless Steels from the 240-hr Nitric Acid Test," p. 68 (TP30); G. W. Jackson and W. A. Luce, "Screening Cast Stainless Steels by Electrolytic Etching in Oxalic Acid," p. 71 (TP33); M. A. Streicher, "Results of Cooperative Testing Program for the Evaluation of the Oxalic Acid Etching Test," p. 63 (TP25).

Another subgroup under the chairmanship of K. M. Huston of the Armco Steel Corp. is engaged in drafting requirements for the boiling acid copper sulfate evaluation test for stainless steels.

The subgroup under the chairmanship of E. G. Holmberg of the Alloy Steel Products Co. expects to present the final draft of the proposed recommended practice for the cleaning and passivation of stainless steel equipment for consideration and appropriate action at the June meeting of the committee.

Subcommittee V on Mechanical Testing (R. Heyer, chairman).—Mr. H. T. Oatman, chairman of the Task Group on tension testing has reported on the cooperative tension tests of Type 301 flat samples, in light and heavy gages, in the annealed, ¼ hard, and ½ hard conditions. The Task Group has been charged with the completion of the present program and reviewing of the entire program, with a view to possibly repeating a part of it and reporting at the June meeting.

The question of need for better definition of the method of determining elongation in certain flat bar products covered by specifications A 276 was referred to the Task Group on tension testing for study and recommendations.

Subcommittee VI on Metallography (R. Franks, chairman).—The activities of this subcommittee have been largely devoted to the preparation of a formal report on metallographic techniques for identification of sigma phase in austenitic chromium-nickel steels. The committee asks permission to publish this report with the annual report of the committee in these *Proceedings*.[6]

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman).—As a result of a number of proposed revisions in Specifications A 276, a Task Group has been appointed to effect those changes. This specification will be continued as a tentative until such time as the work has been completed.

Subcommittee IX on Specifications for Flat Products (G. W. Hinkle, chairman).—A proposed change in the nominal tensile strength value of Type 430 in Specifications A 176 and A 240, Grade D, from 70,000 psi to 65,000 psi minimum, necessitated by change in the annealing practice for this grade aimed to produce soft temper for drawing, has been submitted to letter ballot of the subcommittee.

The subcommittee also approved the classification of the bend test requirements in Specifications A 240.

The subcommittee voted favorably on the proposal to include AISI Type 305 in Specifications A 167 and A 240. Similar action was taken with respect to a suggestion recommending modification of Specifications A 167 and A 177 to conform with AISI standards as regards permissible variations in dimensions and weight, and to include type number designations as per AISI Manual 24 of April, 1950.

W. H. Hodges' task group is preparing a report on the mechanical properties of extra-low-carbon grades 304L and 316L.

Subcommittee XII on Specifications for High-Temperature Super-Strength Alloys (H. D. Newell, temporary chairman) has elected a permanent chairman and more specifically defined the scope and tasks of the subcommittee. The permanent chairman is L. L. Wyman.

A task group under the chairmanship of W. F. Simmons has undertaken to prepare a complete listing of all available super-strength alloys. Upon completion of this list, specifications will be written for one each of the following: (*a*) hot-cold worked, (*b*) precipitation aging, and (*c*) cast super-strength alloys which will be identified only as A, B, and C. Three subgroups have been appointed to draft tentative specifications for these alloys.

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[6] See p. 143.

A second Task Group with L. B. Fonda as chairman has prepared a compilation of the existing aeronautical materials specifications for high temperature alloys of super-strength properties.

This report has been submitted to letter ballot of the committee, which consists of 97 members; 96 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

M. A. CORDOVI,
Secretary.

APPENDIX

EXPERIMENTS ON ETCHING PROCEDURES FOR THE IDENTIFICATION OF THE SIGMA PHASE IN AUSTENITIC CHROMIUM-NICKEL STAINLESS STEELS

(CONDUCTED UNDER SPONSORSHIP OF SUBCOMMITTEE VI)

In response to requests to Committee A-10 by industrial users of stainless steels that it devise a simple etching procedure for identifying the "sigma phase" in these steels, Subcommittee VI on Metallography of A-10 was directed to make a study of various etching techniques to determine if some simple procedure might be developed to accomplish this end. Samples were prepared and appropriately heat treated to produce the sigma constituent as identified by X-ray diffraction measurements, and furnished to various cooperating laboratories for study. The work has been carried out over the period since 1947, during which time many individuals and organizations have contributed their efforts. The cooperating laboratories in the investigation, at which the experimental work was carried out, were the following:

National Bureau of Standards
Chapman Valve Manufacturing Co.
United States Steel Corp.
Midvale Co.
Armco Steel Corp.
Republic Steel Corp.
Union Carbide and Carbon Research Laboratories
Babcock & Wilcox Co.
The International Nickel Co., Inc.
Canadian Department of Mines and Technical Surveys

PREPARATION FOR TESTS

Steels:

Three compositions were chosen for the investigation, that would provide the desired variations in sigma content. Their analyses as reported by three different laboratories are given in Table I.

The steels were made by the United States Steel Corp. (Type 304), and by the Republic Steel Corp., and were finished to size by the Rustless Iron and Steel Division of the Armco Steel Corp. which distributed the samples.

Steel No. 1 was an AISI 304 type which was not expected to develop the sigma phase.

Steel No. 2 was a low-nickel AISI 317 type containing 3.50 per cent molybdenum.

Steel No. 3 was also an AISI 317 type but contained somewhat more nickel than steel No. 2. Although it was originally felt that the increased nickel content might stabilize the austenite and result in freedom from sigma phase, this did not prove to be the case. After annealing, the steel was wholly austenitic and contained no magnetic ferrite but it did undergo sigma transformation on subsequent heating at elevated temperatures.

Approximately 100 lb of each of the three steels in the form of $\frac{3}{4}$-in. diameter centerless-ground bars for Charpy impact test specimens were first water quenched from 2000 F after being held for $\frac{1}{2}$ hr at temperature. In lots of 18 from each of the three steels, the bars were then heated for 25 and 500 hr at each of the temperatures 1200, 1350, and 1500 F and air-cooled. The samples from these bars were tested at room temperature. Six additional bars of each steel were air-cooled from each of the above temperatures after 500 hr holding time and the impact specimens therefrom tested at the temperatures of heat treatment after a 20-min holding period. A series of six bars of each steel in the quench-annealed condition was also included, and the specimens from this group were tested at room temperature. In the case of the bars subjected to the 25-hr holding time the heat treatment was carried out at a single laboratory. For the longer treatments three different laboratories each handled a single temperature.

TABLE I.—CHEMICAL COMPOSITIONS.

	Steel No. 1			Steel No. 2			Steel No. 3		
Laboratory	A	B	C	A	B	C	A	B	C
Carbon, per cent	0.059	0.065	0.057	0.085	0.079	0.081	0.071	0.070	0.067
Manganese, per cent	0.45	0.46	0.46	1.67	1.66	1.66	1.60	1.57	1.60
Silicon, per cent	0.35	0.34	0.34	0.48	0.49	0.48	0.67	0.72	0.69
Chromium, per cent	18.18	18.22	18.19	17.71	17.86	17.73	18.39	18.34	18.30
Nickel, per cent	9.27	9.33	9.33	9.35	9.45	9.45	13.49	13.57	13.48
Molybdenum, per cent	0.03	0.04	0.06	3.51	3.75	3.44	3.51	3.70	3.48
Nitrogen, per cent	...	0.034	...	...	0.054	...	...	0.060	...

Impact Test Specimens:

The impact test specimens were machined to conform to the Charpy keyhole type B specimens of ASTM Methods E 23.[1] Although the preliminary machining of the specimens to the 10-mm square form was carried out by three different groups, the final notching was done by a single shop. In this latter operation the hole was drilled and the notch completed by a saw cut.

Specimens for Other Tests:

Unless specifically stated, all other tests were carried out on sections from the broken impact test specimens.

X-RAY DIFFRACTION STUDIES

Before an accurate interpretation could be placed upon the results of the various tests, or before any unknown metallographic constituent could be positively identified as sigma, the presence or absence of this phase in the samples had to be established. This was done by a study of the X-ray diffraction patterns. Preliminary tests were first carried out at several laboratories. The samples were prepared either by deep etching to remove phases other than sigma from the surfaces to be exposed, or by obtaining a powder residue extracted by electrolysis in a solution that dissolved phases other than sigma.

In addition to sigma one laboratory

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[1] Tentative Methods of Impact Testing of Metallic Materials (Designation: E 23 - 47 T), 1952 Book of ASTM Standards, Part 1. p. 1451; Part 2. p. 1248.

reported the presence in certain of the samples of a phase other than sigma, characterized by a diffraction pattern like that of alpha-manganese. This phase has been reported by Andrews[2] in steels containing chromium, nickel and molybdenum. It is here referred to as "chi phase." Its presence was later confirmed by other laboratories.

TABLE II.—IDENTIFICATION OF SIGMA AND OF CHI PHASES BY X-RAY DIFFRACTION.

Laboratory	Reheated to 1200 F				Reheated to 1350 F				Reheated to 1500 F			
	25 hr		500 hr		25 hr		500 hr		25 hr		500 hr	
	Sigma	Chi	Sigma	Chi	Sigma	Chi	Sigma	Chi	Sigma	Chi	Sigma	Chi
STEEL NO. 2												
No. 1	..	..	t	t	..	..	P	t	..	..	t	P
No. 2	N	N	N	N	?	?	P	P	P	P	t	P
No. 3	?	..	P	..	P	..	P	..	?	..	P	..
No. 4	..	..	t	t	..	..	P	t	..	..	t	P
No. 5	..	..	..	..	..	..	..	..	..	..	..	..
No. 6	N	..	N	..	N	..	P	..	P	..	P	..
No. 7	N	..	N	..	N	..	P	..	t	..	P	..
STEEL NO. 3												
No. 1	..	..	N	N	..	..	t	N	..	..	P	P
No. 2	..	..	N	N	N	N	t	N	N	N	P	P
No. 3	N	..	N	..	N	..	P	..	..	..	P	..
No. 4	..	..	..	..	..	..	t	..	..	..	P	P
No. 5	..	..	..	..	..	..	..	..	..	..	P	P
No. 6	..	..	..	..	..	..	t	..	..	..	P	..
No. 7	N	..	N	..	N	..	t	..	?	..	P	..

t—trace.
N—no indication—not present.
P—present.
?—presence inconclusive.

In the final tests powder residues were employed prepared by extraction with either ferric chloride (electrolytic) or a solution of cupric ammonium chloride and hydrochloric acid. A separation was made of the ferromagnetic from the nonmagnetic particles of steel No. 1 after treatment at 1200 F for 500 hr, and of steel No. 2 after 25 and 500 hr at 1200 F, by passing a magnetized needle through the residue and dislodging the attached particles from the needle. Diffraction patterns from the ferromagnetic particles of steel No. 1 showed strong lines for carbides, weak lines for austenite, and weak unidentified lines. No conclusive indication of lines for delta iron was found. In steel No. 2 after both 25 and 500 hr at 1200 F the diffraction patterns showed strong lines for austenite and ferrite and weak lines for carbides. The results of the tests are shown in Table II.

X-ray diffraction tests of steel No. 1 showed no evidence of the presence of either sigma or chi regardless of the temperature of reheating or the time at such temperature, while in steel No. 3 only sigma was detected after heating at 1350 F and 1500 F for 500 hr with both phases present after 500 hr heating at 1500 F. In steel No. 2 both phases appeared after 500 hr at 1350 F or only 25 hr at 1500 F. One laboratory reported the presence of sigma in diffraction patterns of steel No. 2 after heating to 1200 F for 500 hr and to 1350 F for only 25 hr. This laboratory did not report the presence of chi phase in any of the steels.

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[2] Andrews and Brookes, "Metal Treatment and Drop Forging," July, 1951, pp. 301-311; Andrews, *Nature*, December 10, 1949, p. 1015.

MAGNETIC PERMEABILITY

Preliminary magnetic measurements were made using a Magne-gage, the results of which are shown in Table III.

Precision permeability determinations are given in Table IV. In order to insure that no worked metal was present in the samples in these permeameter tests, sections approximately $\frac{1}{4}$ in. cube were prepared from the end of the specimen away from the fracture and all faces ground to remove all traces of tool and stamping marks. The specimens were then etched with a hot solution of equal parts of concentrated hydrochloric acid and water to remove about 0.005 in. of metal from each face.

TABLE III.—MAGNE-GAGE READINGS.

Condition	Steel No. 1			Steel No. 2		
	Lab. No. 1	Lab. No. 2	Lab. No. 3	Lab. No. 1	Lab. No. 2	Lab. No. 3
2000 F—½ hr, water quenched	0	0	0	2.2	7.8	6
Reheated to:						
1200 F—25 hr	2.3	7	4	2.2	7	6
500 hr	13.3	23.5	24	3.1	8.6	6
1350 F—25 hr	2.9	5.8	3	2.8	7.1	4
500 hr	1.1	5.0	2	0 to 1.4	1.4	0
1500 F—25 hr	0.25	1.0	0	0	1	0
500 hr	0.8	0.8	0	0	1	0

TABLE IV.—MAGNETIC PERMEABILITY MEASUREMENTS (FIELD STRENGTH—436 OERSTEDS).

Condition	Steel No. 1	Steel No. 2	Steel No. 3	Steel No. 2 (directional measurements)[a]	
	μ	μ	μ	μ_P	μ_N
2000 F—½ hr water	1.0028	1.127	1.0028	1.127	1.046
1200 F—25 hr	1.063	1.096	1.0020	1.096	1.040
500 hr	1.223	1.140	1.0027	1.140	1.056
1350 F—25 hr	1.064	1.087	1.0027	1.087	1.028
500 hr	1.0158	1.0025	1.0031	1.0025	...
1500 F—25 hr	1.0057	1.0024	1.0027	1.0024	...
500 hr	1.0036	1.0026	1.0050	1.0026	...

[a] In the case of Steel No. 2 (directional measurements):
μ_P is the permeability observed with field parallel to rolling direction.
μ_N is the permeability observed with field perpendicular to rolling direction.

Samples having permeabilities less than 1.004 mu probably contained no ferromagnetic phase, whereas higher values indicate its presence. Even in the case of the highest values observed (1.22 mu for steel No. 1 after 500 hr at 1200 F) the concentration is probably less than 1 per cent. The data for steel Nos. 1 and 2 are plotted in Fig. 1.

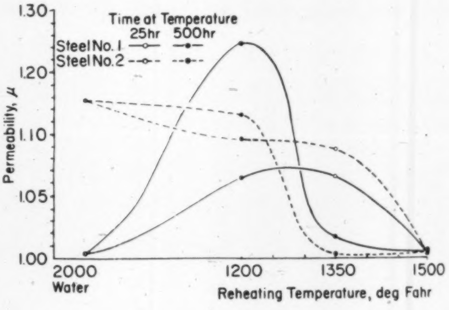

FIG. 1.—Permeability Values for Steels Nos. 1 and 2.

Metallographic examination had shown that the ferrite in steel No. 2 was in the form of "stringers." The four specimens of this steel that showed definite evidence of ferromagnetism were measured with the magnetic field both parallel and perpendicular to the direction of rolling. The lower values at 90 deg to the direction of rolling verified the elongated character of the constituent in the direction of rolling. No such directional effect was found in the permeability values for the other steels.

A consideration of the permeability data indicates a nonmagnetic state in the case of the quench-annealed specimen of steel No. 1, with the development of a magnetic phase after reheating to 1200 F and to 1350 F. At higher temperatures the steel again becomes nonmagnetic. In the case of steel No. 2 transformation from a magnetic to a nonmagnetic condition is indicated after extended heating at 1350 F or after shorter treatment at higher temperature. Steel No. 3 remained nonmagnetic throughout the heating cycles.

TABLE V.—AVERAGE CHARPY IMPACT VALUES, KEYHOLE NOTCH.

Steel	Laboratory	2000 F, Water Quenched	Number of Specimens		Reheated to 1200 F		Reheated to 1350 F		Reheated to 1500 F	
			Water Quenched	Reheated	25 hr	500 hr	25 hr	500 hr	25 hr	500 hr
No. 1	A	65.5	2	5	63.8	57.7	56.9	49.9	55.5	48.7
	B			6	64.4	59.6	56.9	50.6	58.2	50.9
	C			5	80.0	77.8	70.4	63.0	69.4	63.8
	D	57.8	1	1	65.1	54.2	53.3	48.1	54.2	47.3
	Average	62.9		12[a]	64.3	58.3	56.2	49.7	56.5	49.6
No. 2	A	76.3	3	6	49.0	39.2	40.6	18.1	29.8	11.2
	B			6	52.7	42.9	45.0	20.9	30.4	11.7
	C			5	62.0	50.4	50.2	18.6	39.8	11.8
	D	78.4	1	1	49.0	37.3	40.6	18.5	31.1	7.8
	Average	76.8		13[a]	50.7	40.8	42.6	19.4	30.1	11.2
No. 3	A	96.3	3	6	66.6	40.0	41.3	15.4	24.3	4.8
	B			6	70.5	44.2	44.3	17.3	25.4	4.8
	C			5	95.2	50.8	47.8	18.4	30.2	5.0
	D	88.0	1	1	61.3	37.3	41.4	12.9	21.1	3.0
	Average	94.3		13[a]	68.0	41.7	42.7	16.2	24.6	4.8

[a] Results from Laboratory C not included.

There is reason to believe that the permeability of steel No. 2 should increase after exposure to 1200 F for 25 hr as it does in steel No. 1. That it tends rather to decrease suggests the formation of some nonmagnetic sigma even at this low temperature although X-ray diffraction tests generally failed to disclose its presence.

CHARPY IMPACT TESTS

In the first series of Charpy impact tests that were carried out at room temperature, four laboratories participated. The testing machines employed varied in design and make from laboratory to laboratory, but all excepting that used by Laboratory C conformed to the requirements of the Tentative Methods of Impact Testing of Metallic Materials (ASTM Designation: E 23 – 47 T) for Simple Beam Type Apparatus (Charpy Type Test).[1]

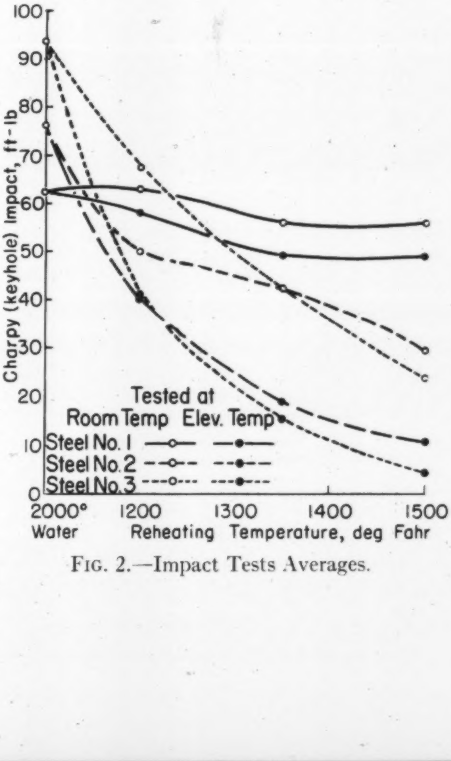

FIG. 2.—Impact Tests Averages.

The results of the tests are presented in Table V and the general averages are illustrated graphically in Fig. 2. In the case of two of the laboratories the values in the table represent averages of six tests (Laboratories A and B), in one an average of five (Laboratory C), and in another a single test (Laboratory D). The data obtained by Laboratory C, due to departure from standard conditions, were considerably higher than those of the other three, as an examination of the table will show, and they were not considered in calculating the general averages.

The loss of notched-bar impact breaking energy in specimens of steel Nos. 2 and 3 is far greater than that found for the plain 18-8 steel No. 1 and indicates that the impact value is more sensitive to the effects of sigma than is any other test with the possible exception of the magnetic test which appears to detect the effects of sigma in steel No. 2 after an exposure of but 25 hr at 1200 F. The magnetic test is, however, useless in the case of steel No. 3 which remains nonmagnetic in all conditions of heat treatment. Although neither X-ray diffraction patterns nor metallographic examination discloses sigma in either steel No. 2 or steel No. 3 after the short exposure at 1200 F, the very pronounced lowering of the impact values appears to be clear evidence of its presence. The marked effect of time at 1200 F further suggests that real sigma embrittlement might ultimately occur at this temperature.

TABLE VI.—AVERAGE CHARPY IMPACT VALUES—KEYHOLE NOTCH (ELEVATED TEMPERATURES).

Steel	Tested after Reheating to 1200 F—500 hr		Tested after Reheating to 1350 F—500 hr		Tested after Reheating to 1500 F—500 hr	
	Room Temp.	At 1200 F	Room Temp.	At 1350 F	Room Temp.	At 1500 F
No. 1	58.3[a]	54.0[b]	49.7[a]	46.3[b]	49.6[a]	52.6[b]
No. 2	40.8[a]	39.6[b]	19.4[a]	28.8[b]	11.2[a]	24.2[b]
No. 3	41.7[a]	50.4[b]	16.2[a]	35.5[c]	4.8[a]	22.8[b]

[a] Room temperature averages are of 13 specimens, 500 hr holding time.

[b] Average of 6 specimens broken at temperatures indicated—previously treated at temperature of testing for 500 hr.

[c] Average of 3 specimens.

Impact Values at Elevated Temperatures:

Tests made at elevated temperatures by Laboratory D gave the results shown in Table VI in which the corresponding room temperature test values are also given. The results are shown graphically in Fig. 3.

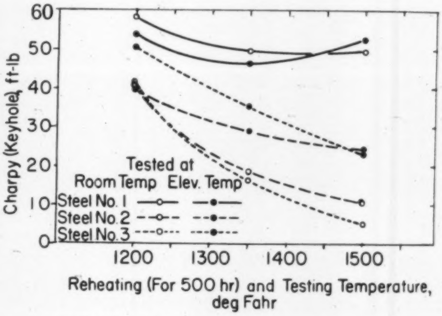

FIG. 3.—Charpy Values at Elevated Temperatures.

As shown in Fig. 2 no marked effect was observed on the impact values at room temperature of steel No. 1 regardless of temperature of reheating or time at temperature; neither was the steel affected materially by the temperature at which the tests were conducted, as shown in Fig. 3.

Table VI shows that Charpy values determined at 1350 and 1500 F were much higher than at room temperature for specimens in comparable condition for steels Nos. 2 and 3.

HARDNESS

One of the laboratories determined microhardness on the banded portion of steel No. 2 (ferrite in the quench-annealed condition) and also on its matrix portion. The tests were made with an Eberbach Microhardness Testing Machine using a 7.5-g load. The results, in D.P.H. (diamond pyramid hardness) units are given in Table VII and are shown diagrammatically in Fig. 4. The latter figure illustrates the point that a marked difference exists between the hardness of the bands and that of the matrix only after reheating to 1350 and 1500 F. It is noteworthy also that the hardness of the matrix does not increase materially with time at temperature, whereas that of the bands does.

TABLE VII.—EBERBACH MICROHARDNESS (D.P.H.), 7.5-G LOAD (STEEL NO. 2).

	2000 F, Water Quenched	Reheated to 1200 F		Reheated to 1350 F		Reheated to 1500 F	
		25 hr	500 hr	25 hr	500 hr	25 hr	500 hr
Matrix	93	145	158	163	177	295	270
Band	91	174	176	221	313	361	440

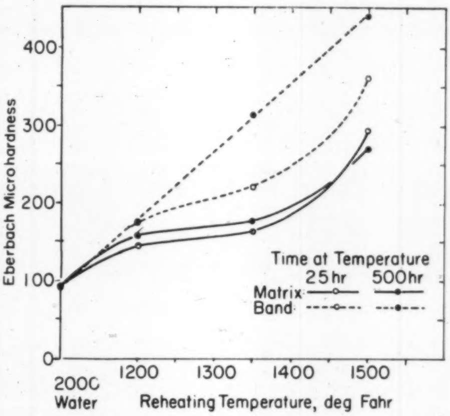

FIG. 4.—Eberbach Microhardness of Steel No. 2.

Two laboratories determined the Rockwell B hardness of the steels in all conditions of heat treatment. These results appear in Table VIII and are illustrated in Fig. 5.

The hardness of steel No. 1 after a 500-hr holding time is lower than for the 25-hr period, whereas this condition is reversed in steel Nos. 2 and 3, particularly at 1350 and 1500 F. The hardness level of steel No. 2 is considerably higher than that of the other two, but the pattern is similar. The pattern resembles that of the other sigma-containing composition, steel No. 3, and conforms to the microhardness trends. There is definite evidence of the increase in amount of a hardening phase, probably sigma, or chi, or both, with increase in holding time at the higher temperature. The microhardness values indicate that most of the increase in hardness of Steel No. 2 is contributed by an increase in the hardness of the bands.

Evidence of the presence of sigma *per se* is not necessarily indicated by an increase in the over-all hardness. For example, steel No. 3 after 500 hr at 1350 F increased to a noteworthy extent, although the amount of sigma present as indicated by the X-ray diffraction tests was but a trace.

The hardness of the sigma particles was reported by one of the laboratories to be of the order of 1980 as compared to 257 for the matrix in steel No. 2.

CORROSION TESTS IN BOILING NITRIC ACID

Samples of the broken impact specimens in the annealed condition and after treatment at the different time periods at 1200, 1350, and 1500 F were properly polished and subjected to the ASTM standard boiling nitric acid test.[3] In these tests the samples were exposed for five consecutive 48-hr periods to boiling 65 per cent acid, and the results are given in Table IX and Fig. 6.

TABLE VIII.—HARDNESS, ROCKWELL B.

	2000 F, Water Quenched		Reheated to 1200 F				Reheated to 1350 F				Reheated to 1500 F			
			25 hr		500 hr		25 hr		500 hr		25 hr		500 hr	
Laboratory No.	1	2	1	2	1	2	1	2	1	2	1	2	1	2
Steel No. 1	82	81.8	77	78.8	76	76.4	79.5	78.3	79	78.4	76	76.1	74	76.2
Steel No. 2	86	86.2	86	85	86.5	87.7	86	87.4	92.5	92.7	89	86.3	94	95.4
Steel No. 3	80	78.2	79	79.3	81.2	79.8	79	77.9	86	89.5	76	76.8	87	87

Readings are averages of 5.

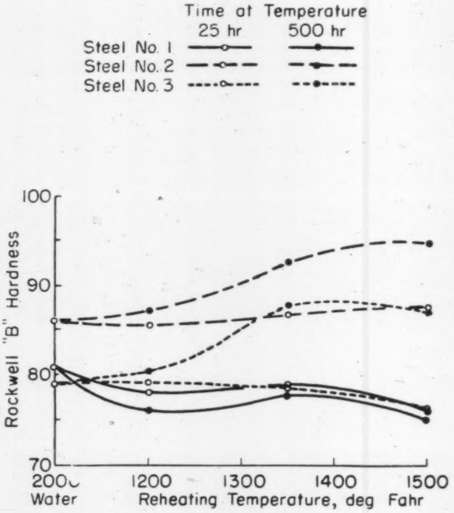

FIG. 5.—Hardness Values.

The corrosion rates given in the table are expressed in inches penetration per month and represent an average of five periods of exposure to the acid. The results show that after heating at 1200, 1350, and 1500 F for either 25 hr or 500 hr all three steels suffered a marked decrease in resistance to the boiling nitric acid. The results further show that all three steels suffered the severest decrease in corrosion resistance after the treatment at 1200 F. Steel Nos. 1 and 2 exhibited a higher degree of corrosion resistance after 500 hr at 1350 F and 1500 F than they did after the treatment for only 25 hr at these temperatures. The opinion can be expressed that the shorter holding period at 1350 and 1500 F renders the steels more subject to corrosion than the longer holding periods because the shorter periods create a very fine precipitation which apparently is more subject to corrosive attack, while the longer holding periods coagulate the precipitate into larger particles that apparently are more re-

TABLE IX.—BOILING NITRIC ACID TESTS.

Identification	Corrosion Rate, in. per month
Steel No. 1-465, ½ hr 2000 F, WQ	0.00080
Steel No. 1-17, 25 hr 1200 F	0.0379
Steel No. 1-125, 500 hr 1200 F	0.0668
Steel No. 1-149, 25 hr 1350 F	0.0186
Steel No. 1-167, 500 hr 1350 F	0.00274
Steel No. 1-191, 25 hr 1500 F	0.00665
Steel No. 1-1109, 500 hr 1500 F	0.00157
Steel No. 2-625, ½ hr 2000 F, WQ	0.00158
Steel No. 2-211, 25 hr 1200 F	0.183
Steel No. 2-227, 500 hr 1200 F	0.137
Steel No. 2-249, 25 hr 1350 F	0.0924
Steel No. 2-267, 500 hr 1350 F	0.0363
Steel No. 2-291, 25 hr 1500 F	0.0174
Steel No. 2-2109, 500 hr 1500 F	0.00757
Steel No. 3-405, ½ hr 2000 F, WQ	0.00145
Steel No. 3-310, 25 hr 1200 F	0.181
Steel No. 3-325, 500 hr 1200 F	0.183
Steel No. 3-349, 25 hr 1350 F	0.136
Steel No. 3-367, 500 hr 1350 F	0.147
Steel No. 3-391, 25 hr 1500 F	0.0292
Steel No. 3-3109, 500 hr 1500 F	0.0904

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[3] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (Designation: A 262 - 52 T), 1952 Book of ASTM Standards, Part 2, p. 998.

sistant to attack by the nitric acid. In the case of steel No. 3 the corrosion rates after the 1200 and 1350 F treatments were considerably higher than after either the 25- or 500-hr treatment at 1500 F, showing that the treatments at 1200 and 1350 F are far more damaging to corrosion resistance than exposure to 1500 F.

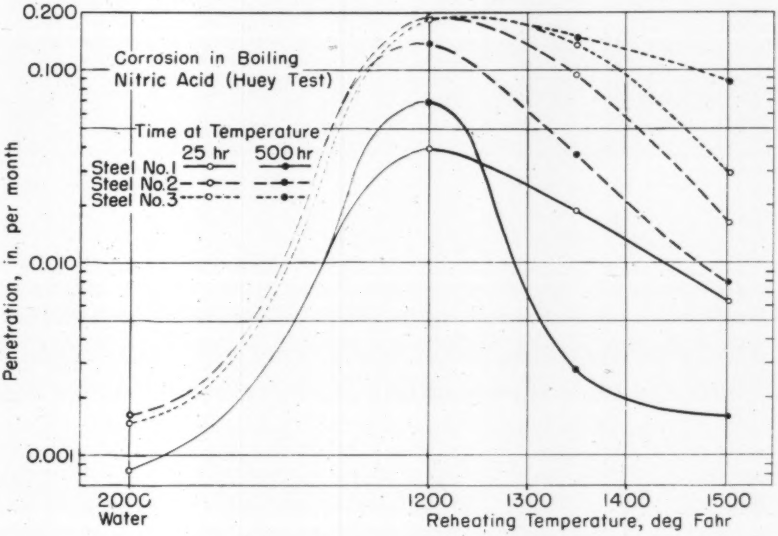


FIG. 6.—Results of Boiling Nitric Acid Tests.

TABLE X.—CHARACTER OF ATTACKS

Heat Treatment			Steel No. 1		Steel No. 2		Steel No. 3	
Deg Fahr	Hr	Quenching Medium	Intergranular	General	Intergranular	General	Intergranular	General
2000	½	Water		Light	Local		Light	Light
1200	25	Air	Severe	...	Severe	Heavy	Severe	Mild
	500	Air	Severe	...	Severe	Heavy	Severe	Mild
1350	25	Air	Severe	...	Severe	Heavy	Severe	Mild
	500	Air	Attack	...	Severe	Heavy	Severe	Mild
1500	25	Air	Attack	...	Attack	Attack	Severe	Moderate
	500	Air	Mild	...	Attack	Attack	Severe	Moderate

An examination of the corrosion test specimens to determine the character of the attack yielded the results shown in Table X.

Generally speaking the greater weight loss of steel Nos. 2 and 3 is accounted for by the heavier "general" attack as distinct from intergranular attack which was suffered in an equal qualitative degree by steel No. 1. The greater loss in weight of steel No. 3 when compared to steel No. 2 appears to be due to more severe grain boundary attack. The "general" attack in the case of steel No. 3 is described as "mild" and "moderate" whereas in steel No. 2 it is "heavy" and just "attack." On the other hand "intergranular" attack is "severe" for both No. 2 and No. 3 after the 1200- and 1350-F exposures and continues to be severe for No. 3 after 1500 F but drops to plain "attack" in No. 2 after 1500-F exposures.

One of the laboratories undertook to examine the specimens after they had been removed from the boiling nitric test for the purpose of determining to what extent either sigma or chi phase was preferentially attacked. The conclusion was reached that the sigma or chi particles that are indefinable in the microstructure of steels Nos. 2 and 3 are not attacked by boiling nitric acid and neither is there evidence that the matrix material immediately adjoining these particles is selectively attacked. It was not possible to determine from microscopical examination whether or not the over-all depletion of the matrix material in chromium due to sigma and chi formation is a factor in corrosion by boiling nitric acid.

Preliminary Metallographic Examination

In the initial metallographic study, the cooperating laboratories were free to choose the etchant or procedure to identify the constituents in the steels. Their individual discussions, and the photomicrographs prepared by them were, therefore, not always comparable. A general summation is given below of the behavior of the various etchants and their indications on the basis of these reported findings. After completion of the preliminary study it was decided to choose a limited number of what appeared to be the most appropriate reagents and to have four different groups each prepare a series of photomicrographs of specimens etched under similar conditions. It would thus be possible to check the reproducibility of any given phenomenon and as a result to more confidently identify the sigma phase.

In the original tests 15 reagents were employed by eight laboratories. In most instances the specimens were polished mechanically, although two groups polished electrolytically. A tabulation of the reagents and the number of laboratories using them are given in Table XI.

TABLE XI.—ETCHING REAGENTS USED.

Etchant	Total No. of Laboratory Users
Electrolytic:	
10 per cent Chromic acid	3
2 per cent Chromic acid	1
10 per cent sodium cyanide	5
10 per cent ammonium persulfate	1
5 per cent nital	1
10 per cent oxalic acid	3
Perchloric acid-alcohol-water	1
Immersion:	
Vilella	5
Marble	1
Murakami (cold)	6
Modified Murakami	3
Mixed acids in glycerine	5
Aqua regia	1

Electrolytic etching with 10 per cent sodium cyanide was the most popula of the electrolytic reagents, while Murakami, used by five groups, was the common choice among those of the immersion type.

Murakami's Reagent:

According to four of the five users this reagent revealed carbides after short etching periods of 5 to 10 sec. With etching times of over 15 sec at room temperature its behavior toward sigma and chi is variable. One user found that it sometimes etched sigma and chi in 20 sec while at other times these phases were not revealed even after 5 min.

Modified Murakami:

This reagent was used by three laboratories. One reported that its action was too rapid when hot to be practical and that it dissolved all of the sigma. Used cold, however, this group found that it colored sigma pastel shades leaving carbides and austenite uncolored. Another found that it developed both sigma and carbides in 30 sec and showed ferrite in steel No. 2. When hot, a 30-sec etch developed sigma that had not been visible after 5 sec in the cold reagent.

Vilella's Etch:

Vilella's etch was used by five laboratories. It was found to outline all phases present. Its use was generally followed by another reagent, after repolishing, to darken either the sigma or the carbides and thus distinguish between them.

Marble's Reagent and Aqua Regia:

These reagents behave generally in a manner similar to Vilella's etch.

Aqua Regia in Glycerine:

Five groups found that this reagent etched grain boundaries and outlined all phases in a manner similar to Vilella's etch.

Electrolytic 10 per cent Sodium Cyanide:

This was the most commonly used reagent of the electrolytic type. Three of the five laboratories found it useful for disclosing the presence of carbides. One employed it as a standard etch for sensitized specimens. It developed grain boundary carbides as well as those within the grain. Two laboratories reported that, after a minute or two, like cold Murakami it colored sigma pastel shades without dissolving it. The reagent outlined and did not stain large carbides but blackened small ones. Ferrite was etched much more rapidly than austenite. One laboratory considered it similar to Vilella's etch when used for less than 3 min. Another used it to identify the high-iron, low-chromium carbides formed at 1200 F, as distinguished from the chromium-rich carbides formed at higher temperatures.

2 per cent Chromic Acid:

One laboratory found that this reagent revealed two constituents in the specimen of No. 3 steel reheated for 500 hr at 1500 F. One constituent was dissolved and the other was outlined.

10 per cent Chromic Acid:

This reagent was used by three laboratories. One reported that it etched out everything, while another reported that it was similar to Vilella's etch. However, examination of their photomicrographs indicated that it dissolved the sigma. A third stated that it attacked both carbides and sigma leaving the ferrite unattacked except when the latter was intimately associated with the sigma, in which event it dissolved both. All considered it most satisfactory for overall study, attacking chromium carbides more readily than carbides rich in iron. In this respect it acted differently from 10 per cent sodium cyanide and enabled a rough estimate to be made of the character of the carbides present.

Oxalic Acid:

Three laboratories reported on this reagent. For short etching times it was similar to Vilella's etch. As in the case of 10 per cent chromic acid the photomicrographs showed that massive sigma was dissolved.

10 per cent Ammonium Persulfate:

One laboratory reported this reagent to behave in a manner similar to the 2 per cent chromic acid.

Electrolytic 5 per cent Nital:

This reagent, in conjunction with aqua regia in glycerine was employed by one laboratory. It did not etch either ferrite or carbides and dissolved out part of the sigma, leaving the remainder unattacked. It was claimed to distinguish sigma from other phases such as chi phase, ferrite and carbides in austenite.

Perchloric Acid:

One laboratory reported that perchloric acid dissolved out sigma progressively with time of treatment.

SUMMARY OF ETCHANTS AND ETCHING EFFECTS

Etching effects appear generally to be of three types:

1. The etchant outlines the phase (aqua regia in glycerine, Vilella's and Marble's).
2. The etchant tints the phase (Murakami, Modified Murakami (cold), electrolytic 10 per cent sodium cyanide).
3. The etchant dissolves out the phase (Modified Murakami (hot), 2 and 10 per cent chromic acid, ammonium persulfate, perchloric acid and electrolytic 5 per cent nital).

The procedure commonly followed in identifying sigma was to use a reagent of Type 1 followed by one of Type 2 or Type 3. Frequently the Type 1 etchant which outlined all the phases without satisfactorily distinguishing among them, was followed by Murakami (Type 2) which darkened the carbides. An etchant of Type 2 used for an appropriate length of time would apparently result in a darkening of the carbides or possibly their removal, with a simultaneous tinting of the sigma phase.

At this point of the investigation a thorough examination was made by Subcommittee VI of all data presented, and it was decided that a limited number of reagents be chosen that had shown promise in the preliminary study and that these be tested by several groups under identical experimental conditions. Four laboratories volunteered for this final work. The detailed procedure followed in preparing the specimens, for example, by the laboratory, and the reagents employed were as follows:

The longitudinal sections of broken impact test specimens were mounted in Bakelite disks $1\frac{1}{2}$ in. in diameter by $\frac{3}{8}$ to $\frac{1}{2}$ in. thick. In order to make electrical contact for electropolishing, a $\frac{1}{8}$-in. diameter hole was drilled into the specimen on the face opposite that to be polished. The surfaces of the specimens were then ground successively with finer abrasive papers ending with the 000 grade. The last two grades were lubricated with a paraffin in kerosine solution.

Polishing was carried through for approximately one minute on a fast 600 alundum wet lap and then on a fine alumina lap. At this stage the specimens were electropolished with a solution of 8.5 ml perchloric acid (70 to 72 per cent), 11.0 ml water, and 80.0 ml alcohol (95 per cent). Optimum voltage and current density were determined by experience. Electropolishing was done in 5-sec intervals, only a slight amount of pitting being obtained. After electropolishing a final mechanical polish with fine alumina was given each specimen.

In order to determine if the disturbed layer caused by grinding had been removed, a back reflection pattern was in each case obtained. This was made with a chromium-target tube. Well-defined spots indicated the absence of such layer. Experience has shown this to be a sensitive test when working with annealed materials. However, a slight amount of disturbance does not affect the appearance of the carbides and sigma phase. If a disturbed layer was indicated, the polishing cycle was repeated until the diffraction pattern showed sharp well-defined spots.

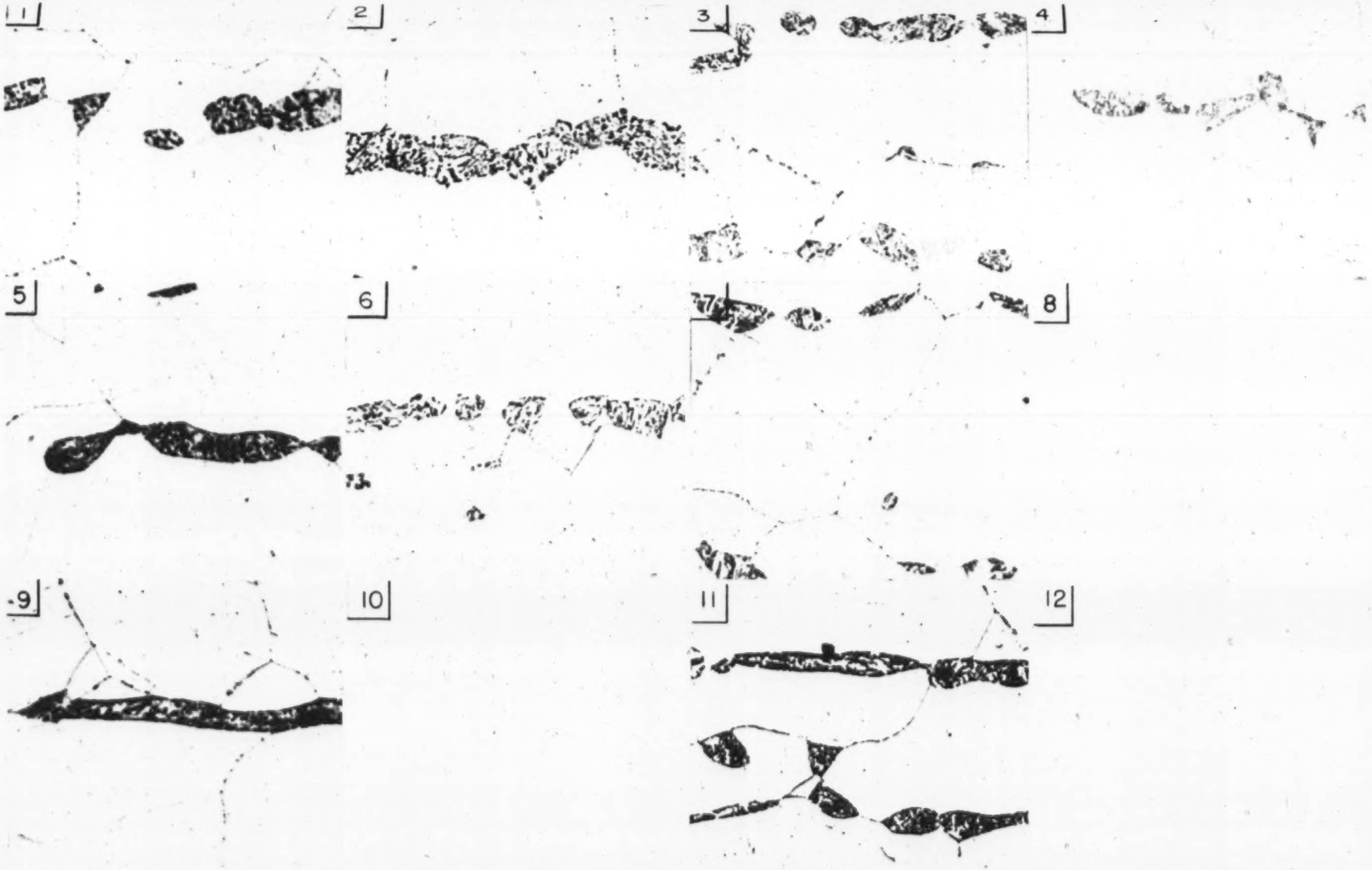
The compositions of the etching reagents, and the times of etch were:

Vilella's Reagent

5 ml Hydrochloric acid
1 g Picric acid
100 ml Ethyl alcohol (95 per cent)
30-sec etching period at room temperature.

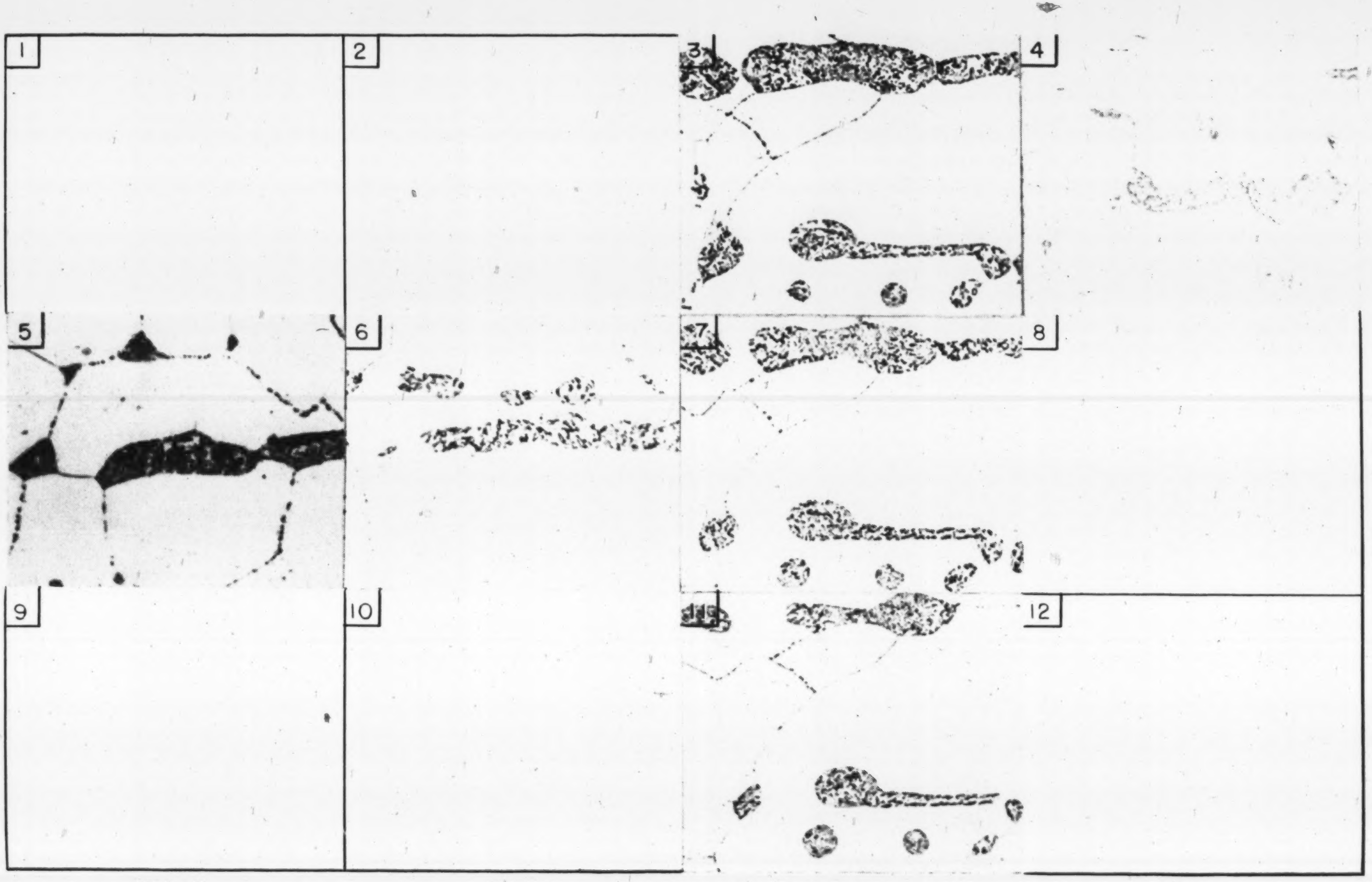
Murakami's Reagent (Dilute)

10 g Potassium ferricyanide
10 g Potassium hydroxide
100 ml Water
5-, 30-, and 120-sec etching periods at room temperature.



Lab. A Lab. B Lab. C Lab. D

Plate A.—Steel No. 2—1350 F—25 hr (× 1500). Reduced to 80 per cent size in reproduction. Etched in 10 per cent NaCN. Top Row—15 sec; Middle Row—60 sec; Bottom Row—120 sec.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Lab. A
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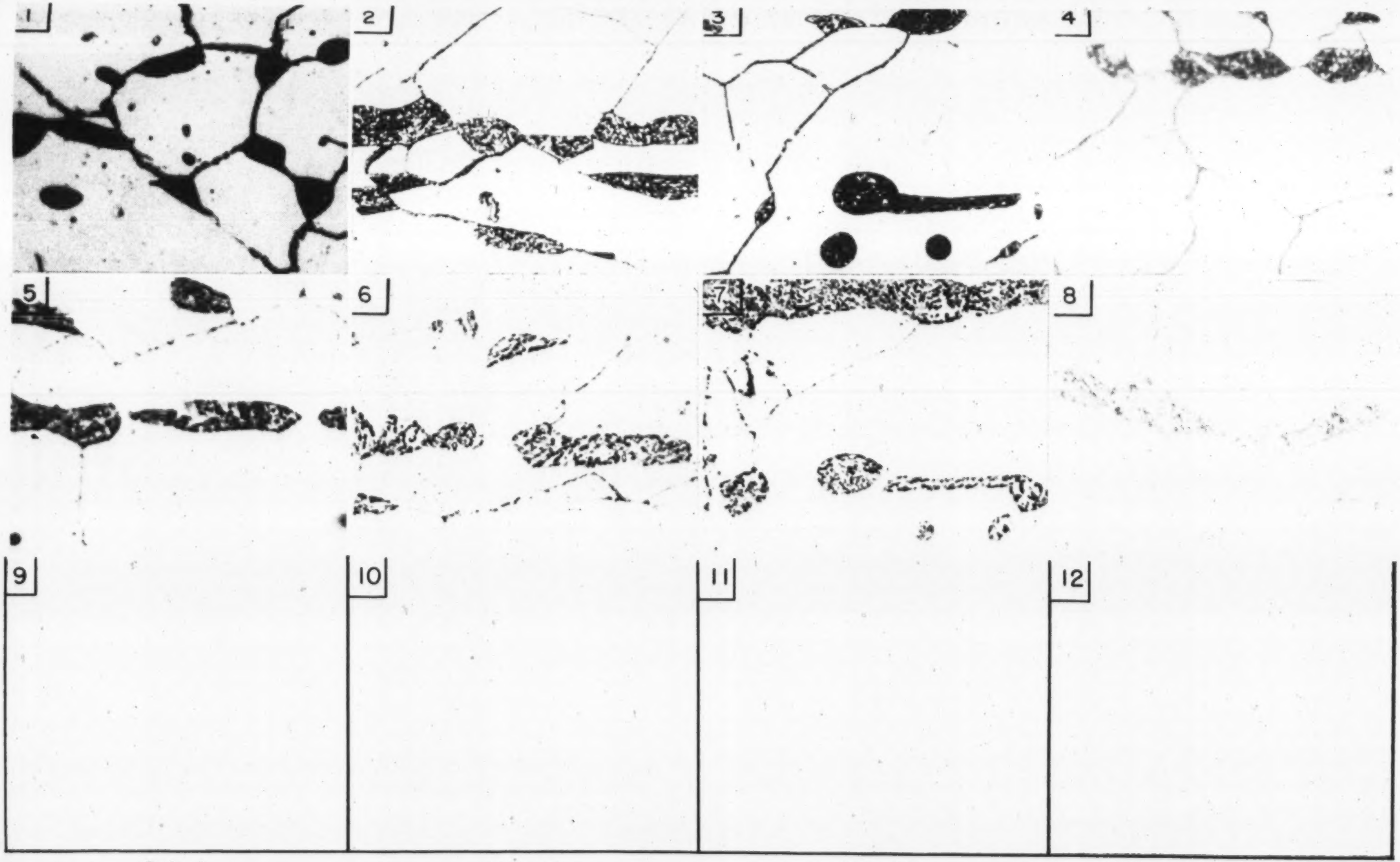
Plate B.—Steel No. 2—1350 F—25 hr (× 1500). Reduced to 80 per cent size in reproduction. Murakami etch.
Top Row—5 sec; Middle Row—30 sec; Bottom Row—120 sec.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Lab. A
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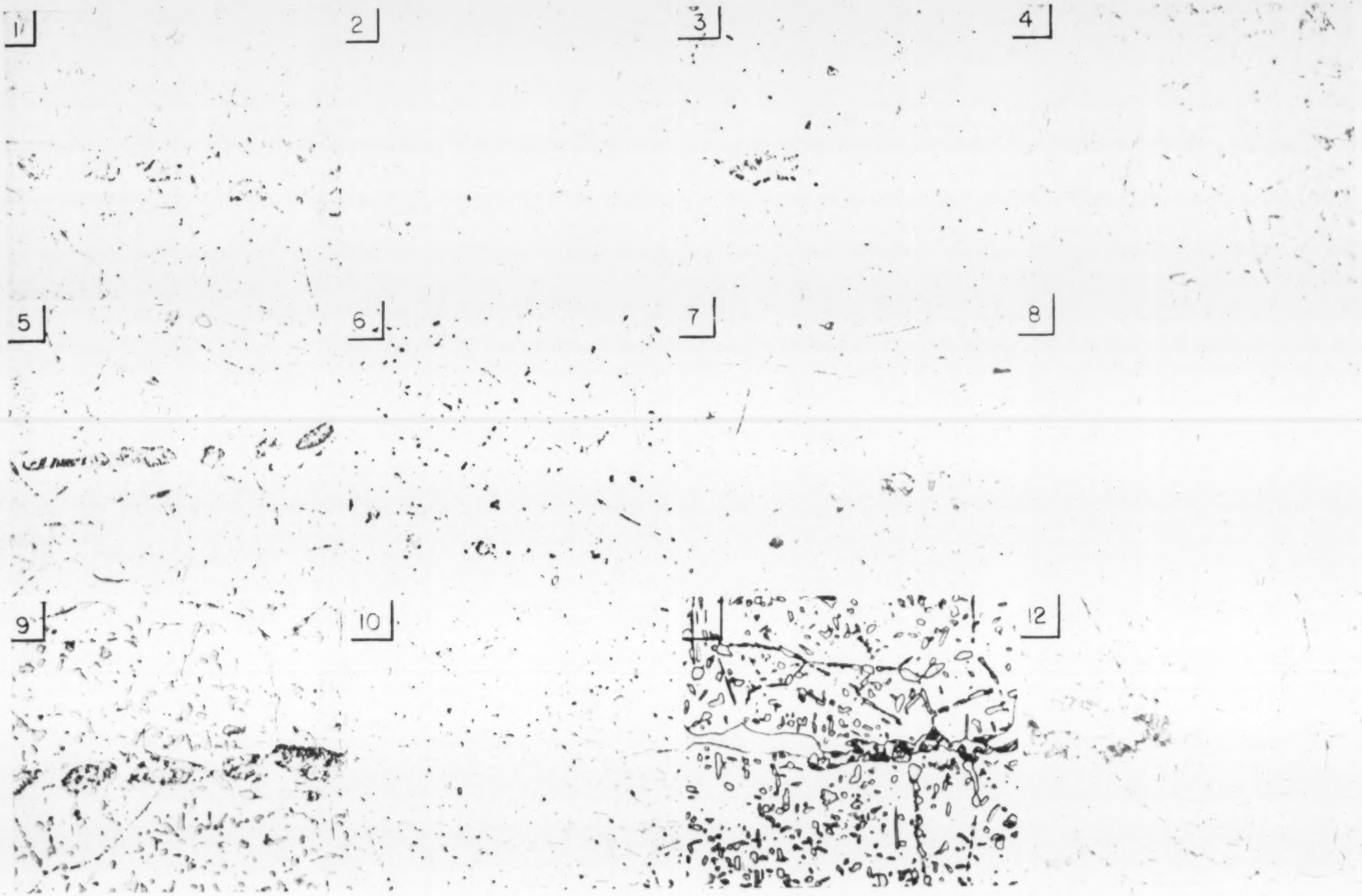
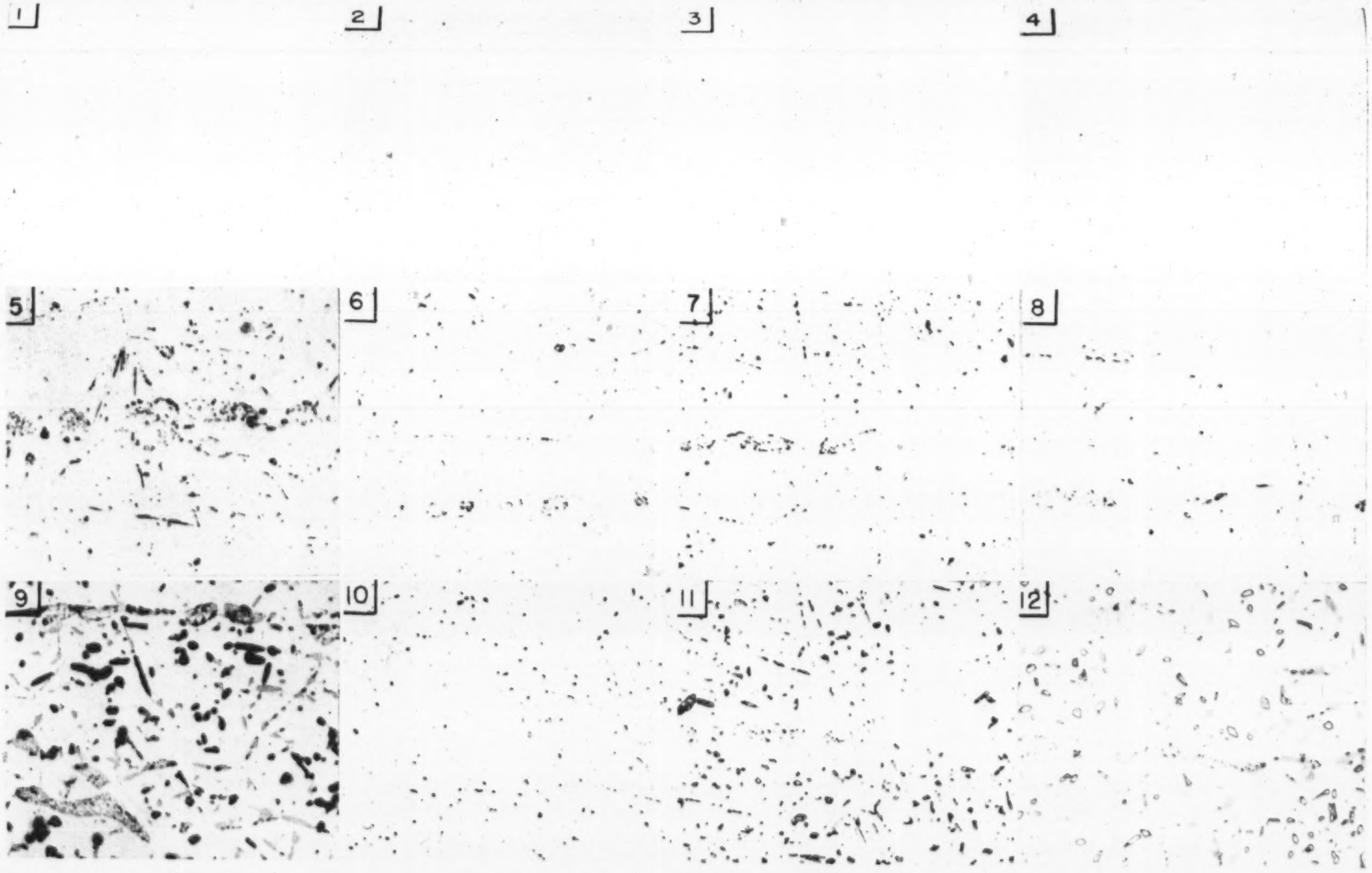
PLATE C.—Steel No. 2—1350 F—25 hr (× 1500). Reduced to 80 per cent size in reproduction.
Top Row—Vilella Etch, 30 sec; Middle Row—Modified Murakami Etch, 5 sec.



Lab. A Lab. B Lab. C Lab. D

PLATE D.—Steel No. 2—1500 F—500 hr (× 1000). Reduced to 80 per cent size in reproduction. Etched in 10 per cent NaCN.
Top Row—15 sec; Middle Row—60 sec; Bottom Row—120 sec.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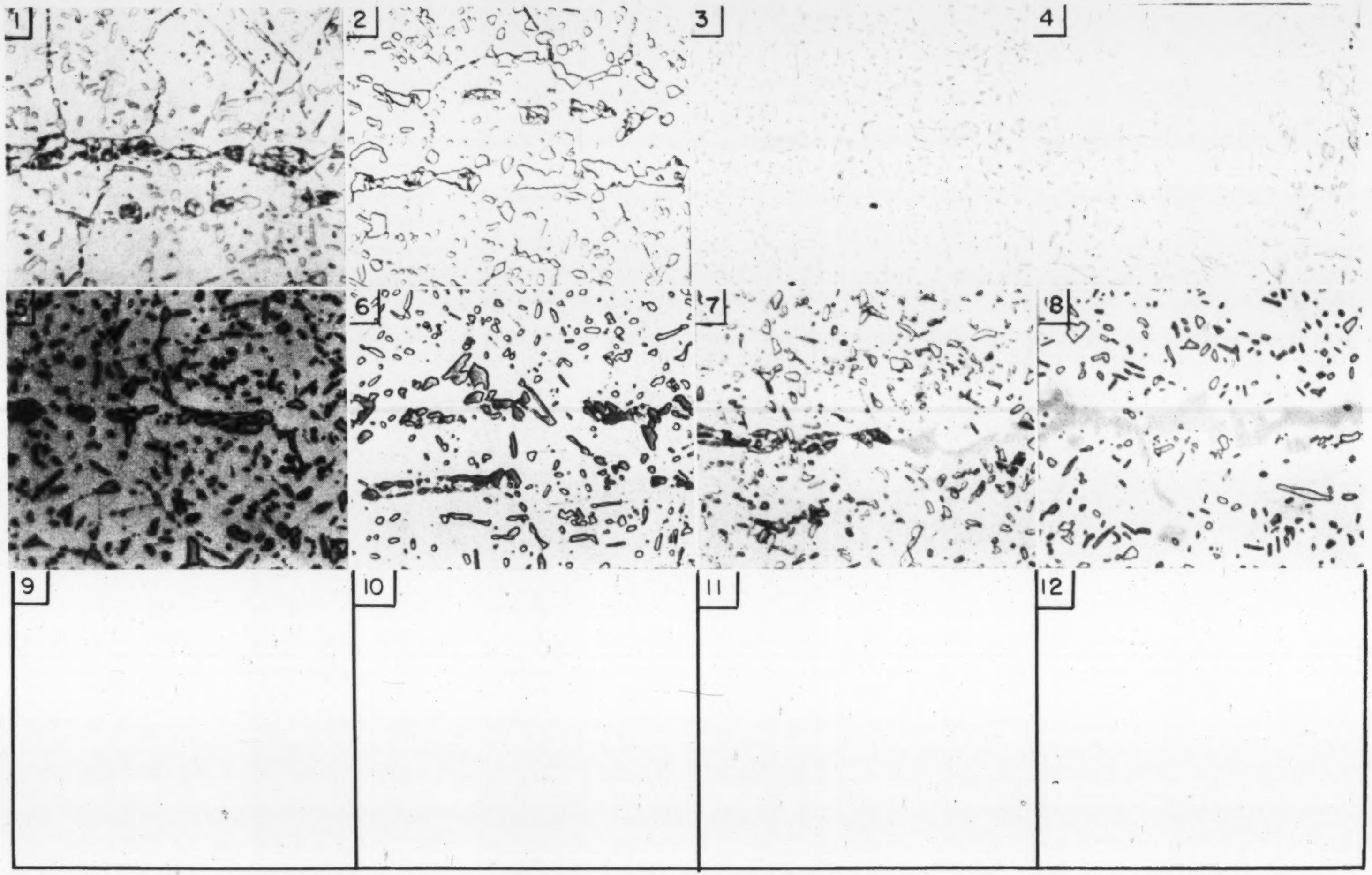
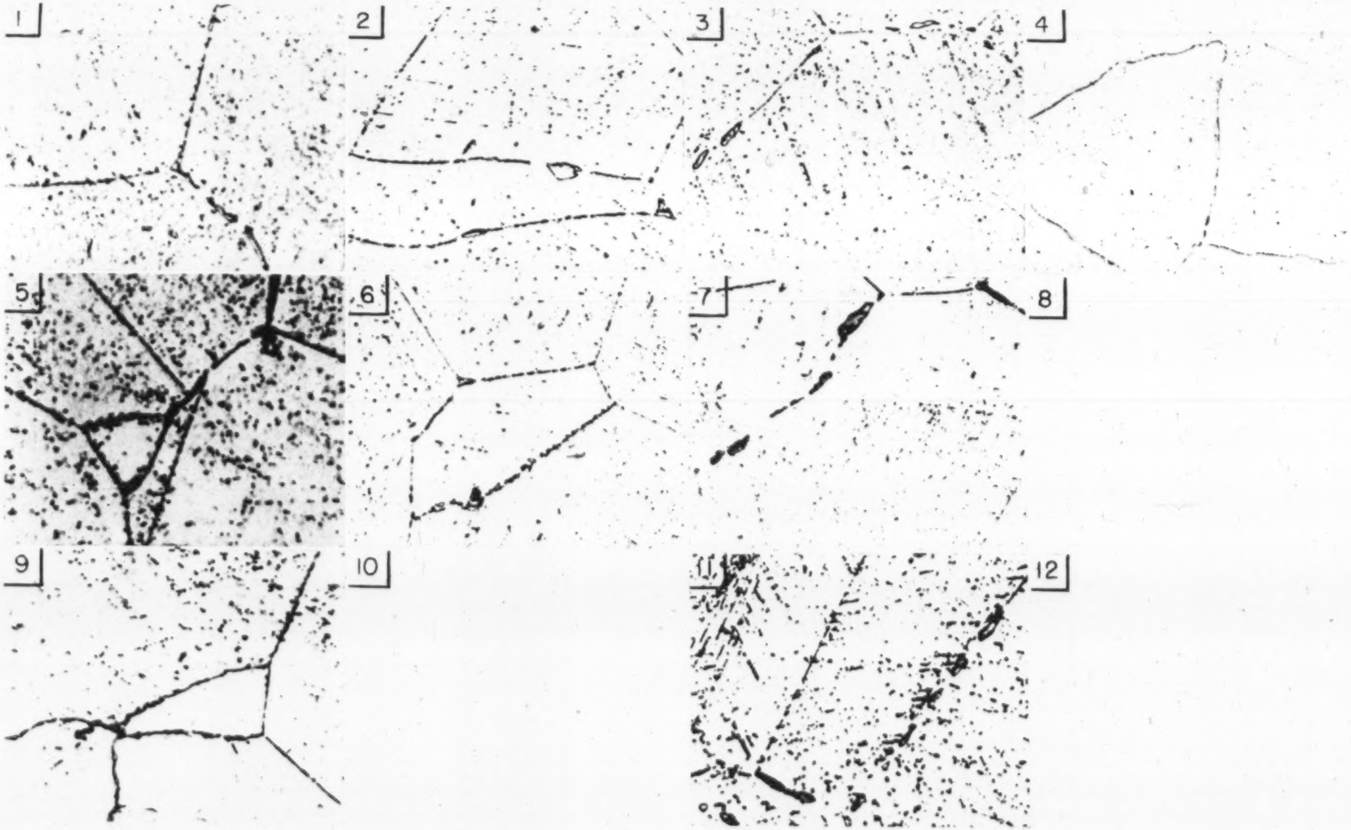
Lab. A Lab. B Lab. C Lab. D

Plate E.—Steel No. 2—1500 F—500 hr (× 1000). Reduced to 80 per cent size in reproduction. Murakami etch.
Top Row—5 sec; Middle Row—30 sec; Bottom Row—120 sec.



Lab. A Lab. B Lab. C Lab. D

PLATE F.—Steel No. 2—1500 F—500 hr (× 1000). Reduced to 80 per cent size in reproduction.
Top Row—Vilella Etch, 30 sec; Middle Row—Modified Murakami Etch, 5 sec.



Lab. A Lab. B Lab. C Lab. D

PLATE G.—Steel No. 3—1350 F—500 hr (× 1500). Reduced to 80 per cent size in reproduction. Etched in 10 per cent NaCN. Top Row—15 sec; Middle Row—60 sec; Bottom Row—120 sec.

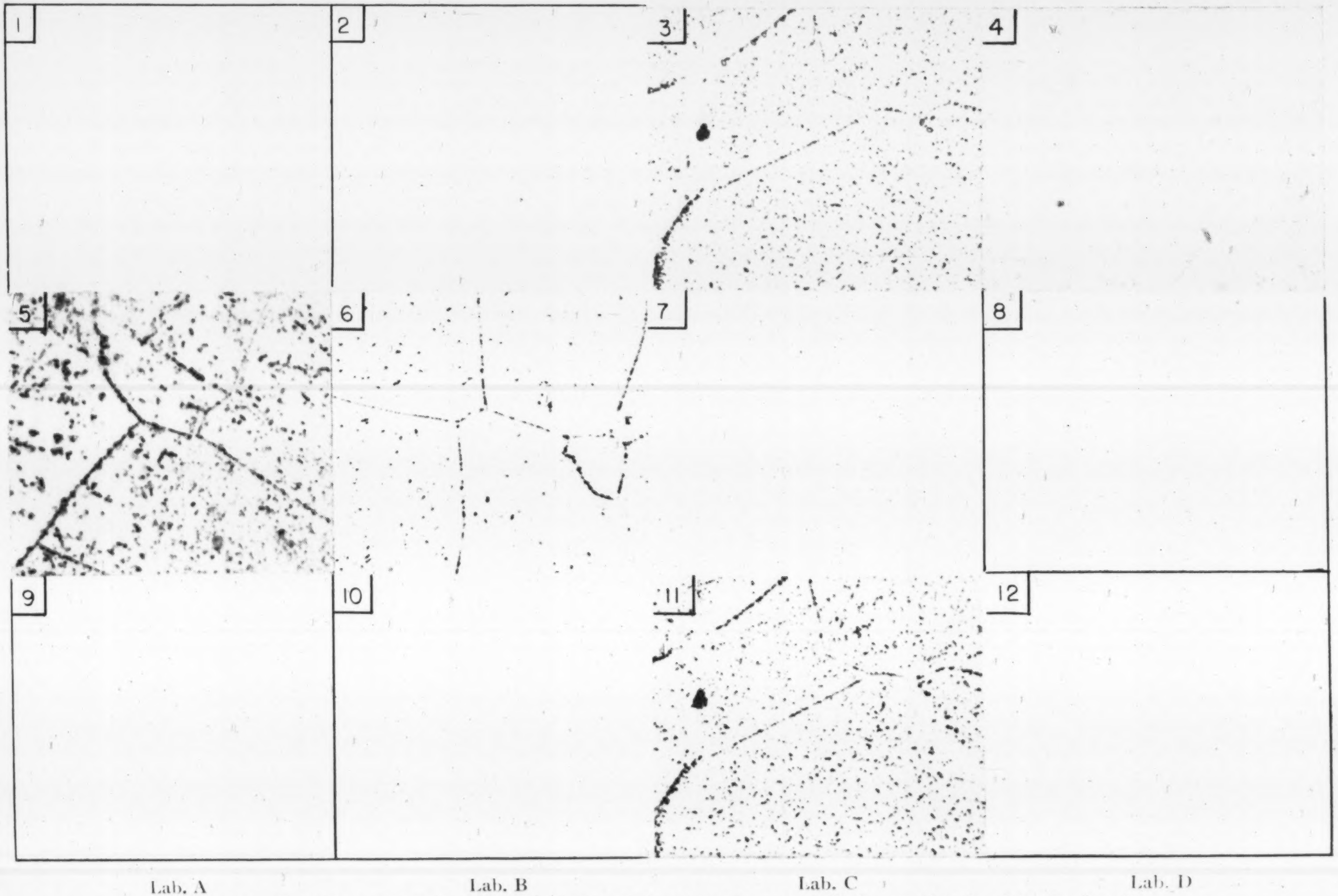

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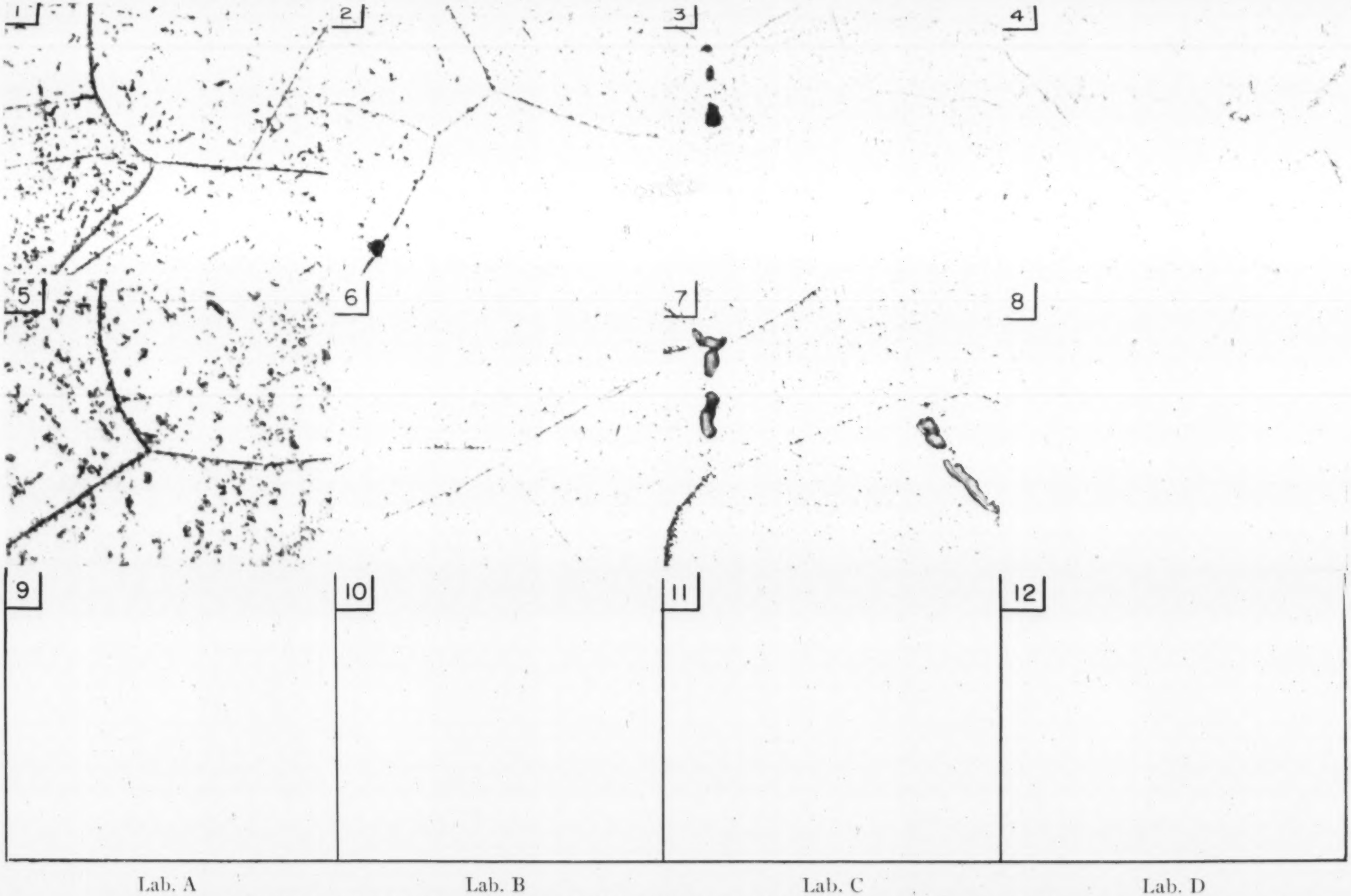
PLATE H.—Steel No. 3—1350 F—500 hr (× 1500). Reduced to 80 per cent size in reproduction. Murakami etch.
Top Row—5 sec; Middle Row—30 sec; Bottom Row—120 sec.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Lab. A
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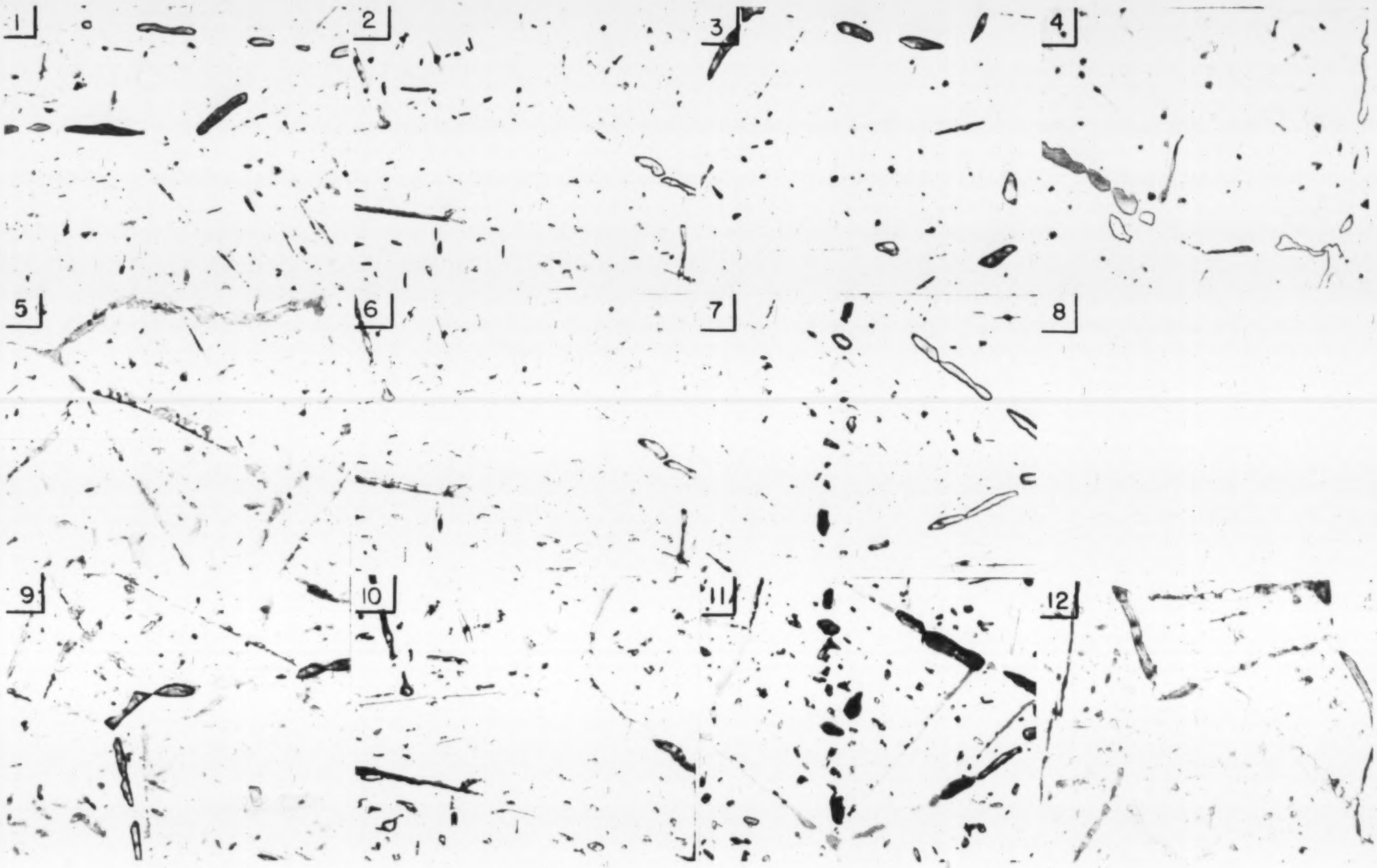
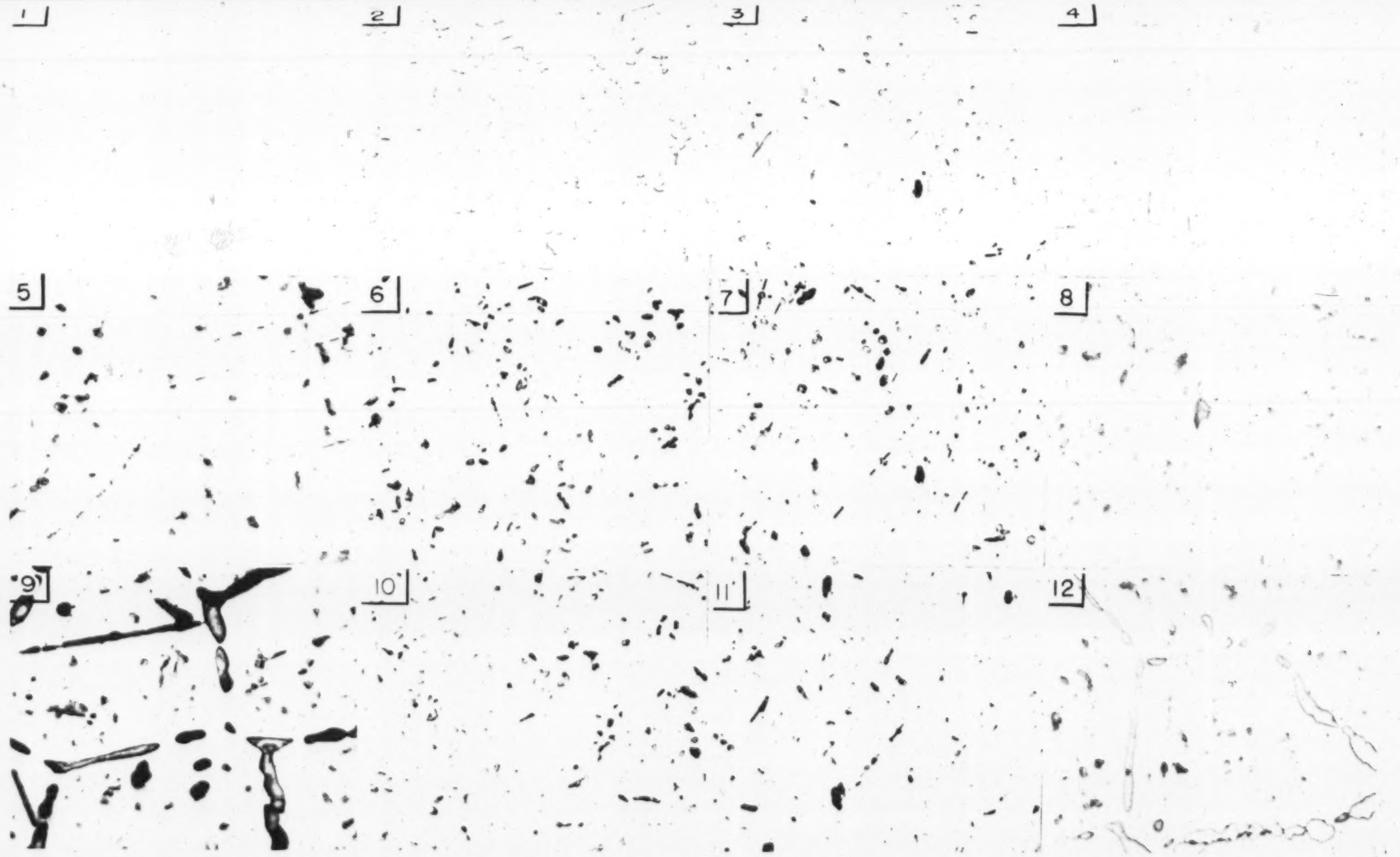
Plate I.—Steel No. 3—1350 F—500 hr (× 1500). Reduced to 80 per cent size in reproduction.
Top Row—Vilella Etch; Middle Row—Modified Murakami Etch, 5 sec.



Lab. A Lab. B Lab. C Lab. D

PLATE J.—Steel No. 3—1500 F—500 hr (× 1000). Reduced to 80 per cent size in reproduction. Etched in 10 per cent NaCN. Top Row—15 sec; Middle Row—60 sec; Bottom Row—120 sec.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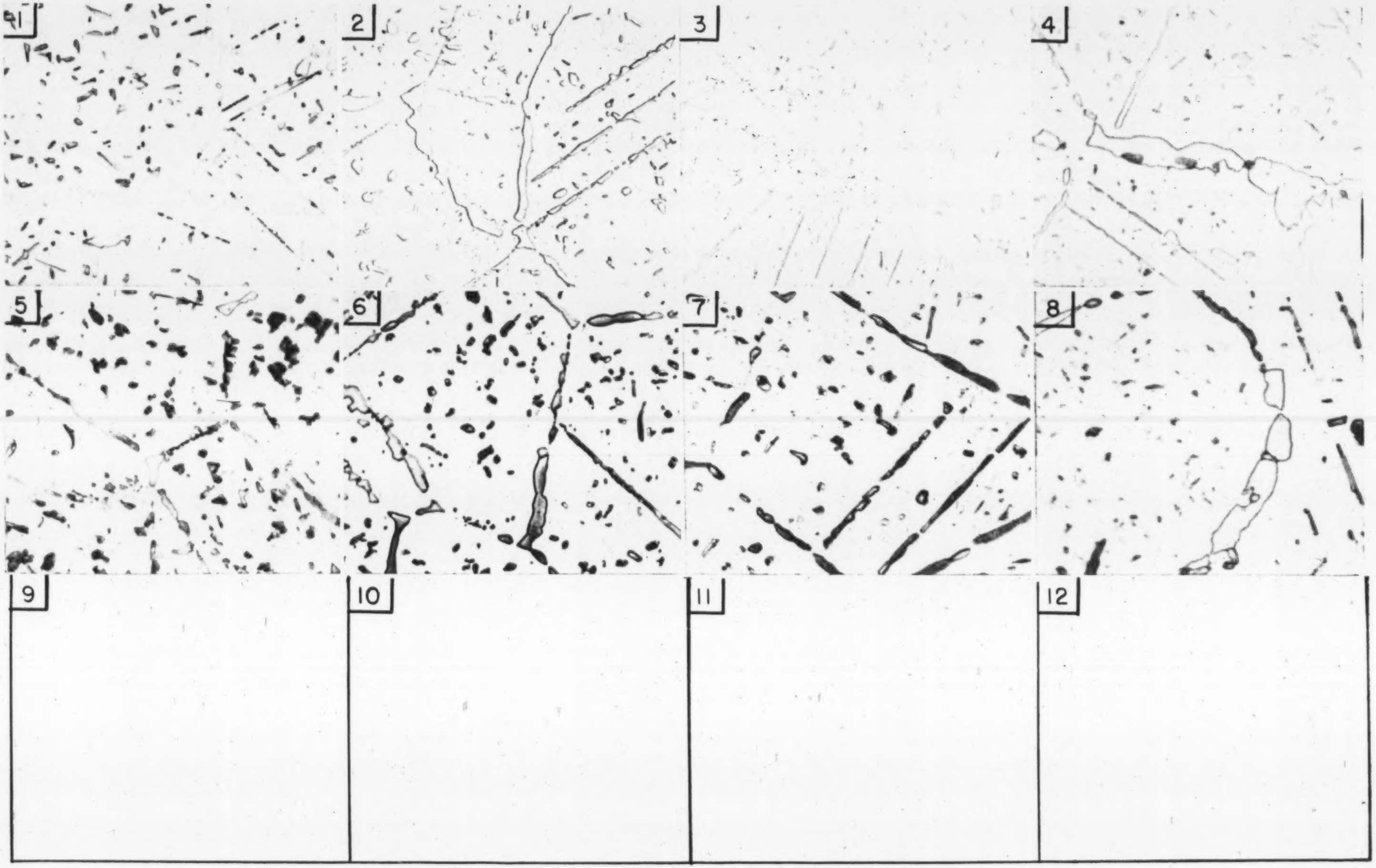
Lab. A Lab. B Lab. C Lab. D

PLATE K.—Steel No. 3—1500 F—500 hr (× 1000). Reduced to 80 per cent size in reproduction. Murakami etch.
Top Row—5 sec; Middle Row—30 sec; Bottom Row—120 sec.



Lab. A Lab. B Lab. C Lab. D

Plate L. Steel No. 3—1500 F—500 hr (X 1000). Reduced to 80 per cent size in reproduction.
Top Row—VilellS etch, 30 sec; Middle Row—Modified Murakami etch, 5 sec.

Murakami's Reagent (Concentrated)

50 g Potassium ferricyanide
50 g Potassium hydroxide
100 ml Water
5-sec etching period at room temperature.

10 per cent Sodium Cyanide (Electrolytic)

10 g Sodium cyanide
100 ml Water
6 v, 0.028 amp per sq cm
¼-, 1-, and 2-min etching periods at room temperature.

Fifty ml of an immersion reagent were made up at a time and used immediately after cooling to room temperature. Fresh solutions were prepared after etching four surfaces. Slightly more solution was necessary for conveniently handling the sodium cyanide reagent.

An attempt was made to photograph the same area after repolishing and etching for different periods with the same reagent. This technique was not always practical, however, because of changes in details of the structure. The structures after the 1350 F and 1500 F anneals were photographed at 1500 and 1000 magnifications respectively. The numerical apertures of the objectives were 0.95 for 1000× and 1.40 oil for 1500×.

For convenience in examination, the photomicrographs are arranged in Plates A to L by heat treatment and reagent types, comparable illustrations by the four cooperating laboratories being presented horizontally.

Vilella's Reagent (Plates C, F, I, and L):

This reagent apparently develops delta ferrite, carbides, and sigma and chi phases when present. However, these constituents cannot be distinguished one from the other except on the basis of distribution, size and shape. The mottled appearance of the "delta ferrite site" in steel No. 2 after 25 hr at 1350 F (Plate C-3) is interpreted as showing a transitory stage in the decomposition of the delta ferrite. The mixture probably consists of austenite, ferrite, carbide, and probably sigma and chi phases. The small particles in Plate I-3 are believed to be carbides. The large light gray areas in the latter plate are sigma or chi phases. The structure shown in Plates F-3 and L-3 appears to be a mixture of carbides and sigma or chi phases, or carbides, sigma and chi phases.

Murakami's Reagent (Plates C, E, H, and K):

The development of carbides after the 1350-F exposures of steel Nos. 2 and 3 was independent of concentration or of etching time. This is apparent from Plates B and H. Two minutes (Plate H-11) were not sufficient for the dilute reagent to develop the structure in steel No. 3 (1350 F – 500 hr) that appeared after 5 sec (Plate I-7) with the modified reagent. Although Laboratories A and D, for some unknown reason, were able to bring out the structure in 120 sec. with the Murakami reagent in Plate E (9 and 12) and Plate K (9 and 12), concentration is shown to be important in both steel Nos. 2 and 3 after 1500 F for 500 hr as shown in Plates E and F and K and L. The complete structure is apparently developed in 5 sec with the "concentrated" reagent (Plates F and L). On the basis of X-ray diffraction findings the fully developed structures brought out by this modified Murakami reagent show, in Plate F, chi phase outlined, the light gray areas being possibly sigma phase particles and in Plate L, both chi and sigma phases, the outlined particles presumably being chi phase and the gray mottled particles sigma phase.

Electrolytic 10 per cent Sodium Cyanide Reagent (Plates A, D, G, and J):

Examination of the structures developed in steel Nos. 2 and 3 after 500 hr at 1500 F (Plates D & J) showed that the two constituents which developed first were below the matrix level, whereas that which developed later was above. X-ray diffraction data had shown that the predominant constituent in steel No. 2 after 500 hr at 1500 F was chi phase and that steel No. 3 after 500 hr at 1500 F definitely contained both sigma and chi phases. From these results it is concluded that the carbide and sigma phases develop first and chi phase second. It follows then, that the structure developed in steel No. 3 after 500 hr at 1350 F (Plates G-3, G-7, G-11) shows the distribution of the carbides and sigma phase. The dot-like particles are carbides.

The darkening of the delta ferrite sites (Plates A-3, A-7, A-11) was given the same interpretation as that for the darkening obtained with the Vilella's reagent. It was pointed out that the outlined particles in the grain boundaries in Plate D probably were chi phase. This conclusion was based on the absence of similar particles in the structures developed by the Murakami reagents (Plate E). The chi phase develops very slowly with these reagents.

ADDITIONAL TESTS

Additional tests were made by T. V. Simpkinson, Canadian Department of Mines and Technical Surveys. In these tests the complete set of corrosion-tested samples was examined to determine whether a relationship exists between the path of corrosive attack and the location of metallographically identifiable sigma or chi particles. These results are appended to this report.[4]

SUMMARY

The results of metallographic examinations made during this investigation show that the sigma phase that develops in the austenitic stainless steels cannot be identified with certainty by the use of an etching reagent alone. The sigma phase can be identified metallographically on properly etched samples provided the particles are large enough, and the composition and thermal heat treatment of the austenitic chromium-nickel stainless steel are known. In other words this work has shown that once the presence of the sigma phase has been established through X-ray diffraction technique in austenitic chromium-nickel stainless steels of certain compositions and certain thermal treatments it can be detected by etching with the proper reagents.

The etching reagents that will outline the sigma phase are *aqua regia* in glycerine, Vilella's, and Marble's reagent. Murakami's and the electrolytic 10 per cent sodium cyanide reagents will tint the phase. The etchants that dissolve the phase are hot modified Murakami's solution, 2 and 10 per cent chromic acid, ammonium persulphate, perchloric acid and the electrolytic 5 per cent nital solution.

The procedure that can be used for identifying the sigma phase under known conditions of composition and thermal treatment of austenitic stainless steels is to use *aqua regia* in glycerine, Vilella's, or Marble's reagent followed by Murakami's reagent either as such or modified, electrolytic 10 per cent sodium cyanide, 2 and 10 per cent chromic acid, ammonium persulphate, perchloric acid or the electrolytic 5 per cent nital etch. Further details of identifying the sigma phase may be found under the "Summary of Etchants and Etching Effects."

Other facts that appear to be established under the conditions of these tests are as follows:

1. The sigma phase does not develop in the low carbon 18-8 steel under the conditions of thermal treatment herein studied. Sigma phase does develop in the molybdenum-bearing austenitic chromium-nickel steels after heat treatment involving temperatures of 1350 and 1500 F.

2. Development of sigma phase in the molybdenum-bearing austenitic chro-

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[4] See p. 170.

mium-nickel stainless steels brings about a marked decrease in the toughness of the steels. The toughness of the 18-8 steel also decreased somewhat by holding at temperatures of 1200, 1350 and 1500 F, but this slight drop in toughness may be accounted for by the presence of precipitated carbides.

3. The most sensitive indication of the presence of sigma was the loss of toughness in the Charpy Impact Test.

4. The absence of sigma in X-ray diffraction patterns obtained by methods used in this investigation or in structures developed by the wide variety of etching reagents used appears to be no sure indication that this phase is not present.

5. The presence of sigma phase brings about an increase in hardness in the segregated bands of transformed alpha phase.

6. The sigma phase identified in the molybdenum-bearing stainless steels is non-magnetic in character.

7. A new phase designated as chi was detected by X-ray along with the sigma phase in the molybdenum-bearing stainless steels. This new phase was not found to exist in the austenitic chromium-nickel steel containing no molybdenum.

Acknowledgment:

Subcommittee VI wishes to express its appreciation to Committee A-10 as a whole for its interest and help in completing the investgiation covered by this report. The chairman and secretary of the subcommittee also wish to express appreciation to certain members of Subcommittee VI who contributed to the successful completion of the investigation. It is appropriate therefore to cite the following for their help:

Jerome Strauss, Vanadium Corporation of America
J. J. Heger, United States Steel Corp.
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S. Low, Chapman Valve Manufacturing Co.
H. C. Vacher, Bureau of Standards
L. L. Wyman, Bureau of Standards
R. H. Heyer, Armco Steel Corp.
M. Clogg, Armco Steel Corp.
G. N. Goller, Armco Steel Corp.
V. N. Krivobok, The International Nickel Co., Inc.
F. L. LaQue, The International Nickel Co., Inc.
T. V. Simpkinson, Canadian Department of Mines & Technical Surveys
W. D. Forgeng, Union Carbide & Carbon Research Labs.
H. D. Newell, Babcock & Wilcox Co.
G. N. Emmanuel, Babcock & Wilcox Co.
G. M. Riegel, Republic Steel Corp.
M. H. Brown, E. I. duPont deNemours & Co., Inc.
J. J. Egan, Electro Metallurgical Co.

Respectfully submitted on behalf of Subcommitte VI,

RUSSELL FRANKS,
Chairman.

FRANCIS B. FOLEY,
Secretary.

[See discussion, page 177]

APPENDIX

METALLOGRAPHIC EXAMINATION OF SAMPLES AFTER BOILING NITRIC ACID TEST*

By T. V. Simpkinson[1]

The Engineering Research laboratory of E. I. du Pont de Nemours and Co. subjected a complete set of impact test sections from the Subcommittee VI sigma phase investigation to the standard boiling nitric acid test. Their reported test results are given below. They judged the type of attack by examination of the corroded surfaces under a binocular microscope.

The du Pont report points out that, where corrosion is shown as intergranular and general, there was corrosion of the grain faces as well as the grain boundary areas.

The author examined the complete set of corrosion tested samples to determine whether a relationship exists between the path of corrosive attack and the location of metallographically identifiable sigma or chi particles. Selected samples were sectioned longitudinally and half sections were mounted in bakelite so that the longitudinal saw-cut face could be polished. Steel strips were placed near the specimen edges to minimize rounding.

Macroexamination

In the case of all samples that suffered relatively heavy corrosive attack, the end faces (transverse section) were corroded more heavily than the longitudinal surfaces. Corrosion of the end faces proceeded faster in some areas than in others. Figure 7 illustrates this condition. There was nothing in the microstructure to explain why some areas of the end faces were attacked faster than other areas.

TABLE XII.—NITRIC ACID TEST RESULTS.

Heat Treatment	Corrosion Rate, in. per month	Type of Attack
Steel No. 1 (Type 304)		
½ hr 2000 F, WQ	0.00080	Light general etching
25 hr 1200 F	0.0379	Severe intergranular attack
500 hr 1200 F	0.0668	Severe intergranular attack
25 hr 1350 F	0.0186	Severe intergranular attack
500 hr 1350 F	0.00274	Intergranular attack
25 hr 1500 F	0.00665	Intergranular attack
500 hr 1500 F	0.00157	Mild intergranular attack
Steel No. 2 (Type 317, Low Ni)		
½ hr 2000 F, WQ	0.00158	Local intergranular attack
25 hr 1200 F	0.183	Severe intergranular and heavy general attack
500 hr 1200 F	0.137	Severe intergranular and heavy general attack
25 hr 1350 F	0.0924	Severe intergranular and heavy general attack
500 hr 1350 F	0.0363	Severe intergranular and heavy general attack
25 hr 1500 F	0.0174	Intergranular and general attack
500 hr 1500 F	0.00757	Intergranular and general attack
Steel No. 3 (Type 317)		
½ hr 2000 F, WQ	0.00145	Light grain boundary and general etching
25 hr 1200 F	0.181	Severe intergranular and mild general attack
500 hr 1200 F	0.183	Severe intergranular and mild general attack
25 hr 1350 F	0.136	Severe intergranular and mild general attack
500 hr 1350 F	0.147	Severe intergranular and mild general attack
25 hr 1500 F	0.0454	Severe intergranular and moderate general attack
500 hr 1500 F	0.0904	Severe intergranular and moderate general attack

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Division of Physical Metallurgy, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Ont., Can.

MICROEXAMINATION

Etchant Used:

For all photomicrographs in this report, the specimens were etched electrolytically in 10 per cent sodium cyanide for 30 sec at 6 v.

Steel No. 1:

No sigma phase or chi phase was found in any of the specimens of steel No. 1. Examination of the corroded test pieces showed that corrosive attack was intergranular. The manner of attack is illustrated in Fig. 8 (sample heated at 1200 F for 500 hr). It is evident that little if any attack upon the grain faces has occurred.

Steel No. 2:

Steel No. 2 "as quench-annealed" contained an appreciable amount of ferrite, present as longitudinal "stringers." When this steel is reheated in the sigma forming temperature range, the first place where sigma should form is in the ferrite sites. The corrosion specimens were examined to see if selective attack upon these ferrite sites had occurred. The following was observed:

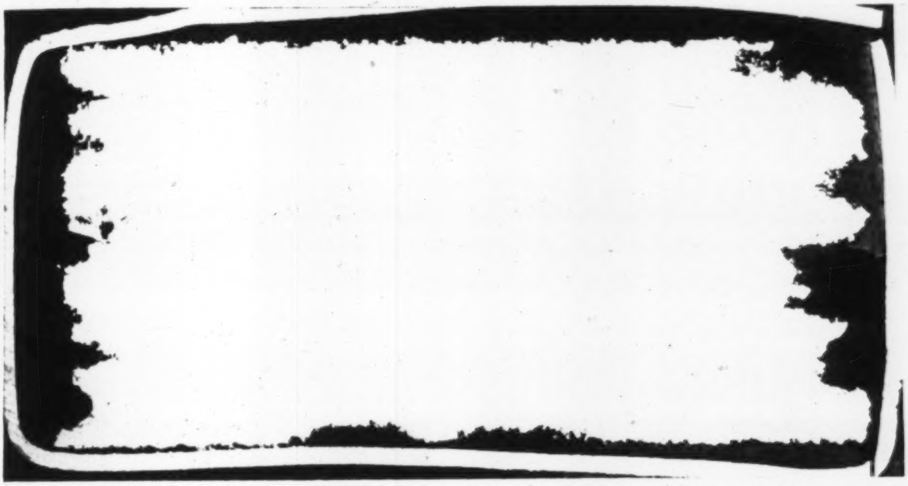


FIG. 7.—Steel No. 3—1500 F—500 hr. Corrosion Rate, 0.0904 ipm (× 6).

Treatment	Evidence of Selective Attack
1200 F 25 hr	Some
1200 F 500 hr	Pronounced
1350 F 25 hr	Some
1350 F 500 hr	None
1500 F 25 hr	Some
1500 F 500 hr	None

The manner of attack on the 1200 F – 500 hr sample is illustrated in Fig. 9. As well as attack on the ferrite sites, there was heavy grain boundary attack and mild general attack. A trace of both sigma and chi was detected in this sample by means of X-ray

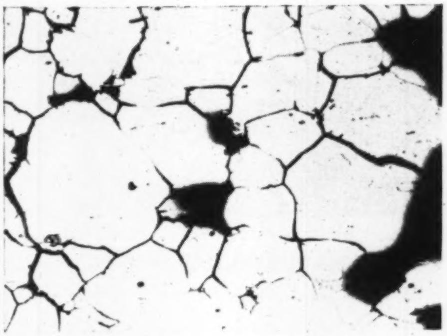
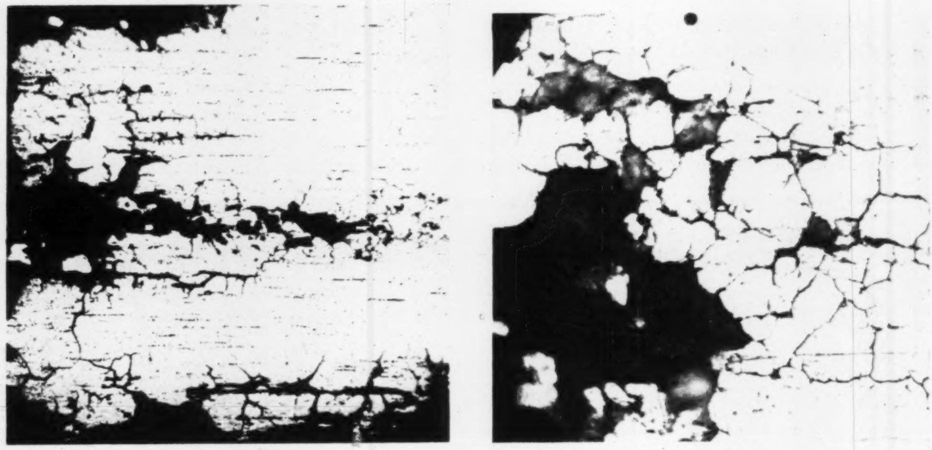


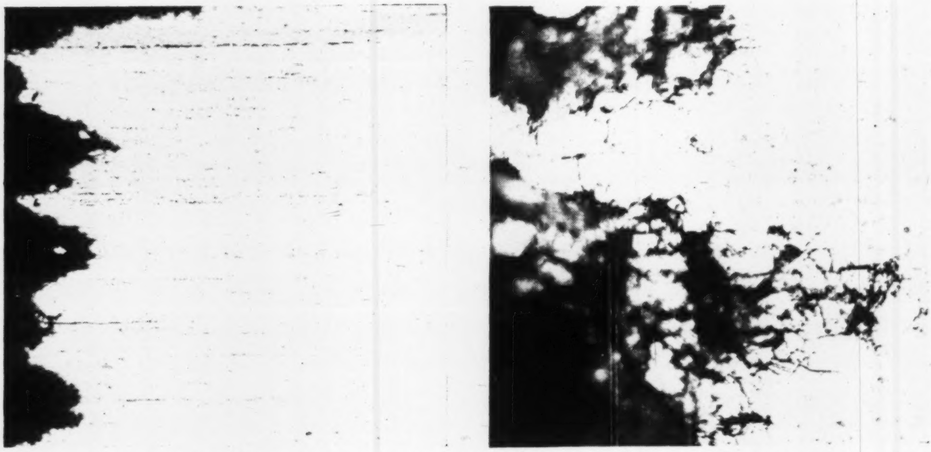
FIG. 8.—Steel No. 1—1200 F—500 hr. Corrosion Rate, 0.0668 ipm (× 100). Reduced to ⅔ size in reproduction.

diffraction, but it was not possible to identify these phases in the microstructure.



(*a*) (× 50). Reduced to ⅔ size in reproduction. (*b*) (× 250). Reduced to ⅔ size in reproduction.

FIG. 9.—Steel No. 2—1200 F—500 hr. Corrosion Rate, 0.137 ipm.



(*a*) (× 50). Reduced to ⅔ size in reproduction. (*b*) (× 250). Reduced to ⅔ size in reproduction.

FIG. 10—Steel No. 2.—1350 F—500 hr. Corrosion Rate 0.0363 ipm.

With the 1350 F - 500 hr sample there was no pronounced attack upon the former ferrite sites, and general attack was so rapid that intergranular attack was largely obscured (see Fig. 10). X-ray diffraction studies showed that both sigma and chi phases were present in this sample. A constituent in the microstructure that is sigma or chi or both is easily recognized, and microscopic examination of the corrosion test specimen showed

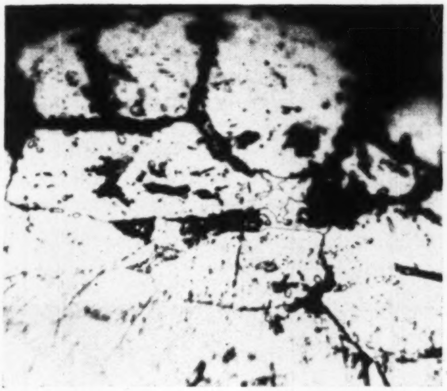


FIG. 11.—Steel No. 2—1350 F—500 hr. Corrosion Rate, 0.0363 ipm (× 1000). Reduced to ⅔ size in reproduction.

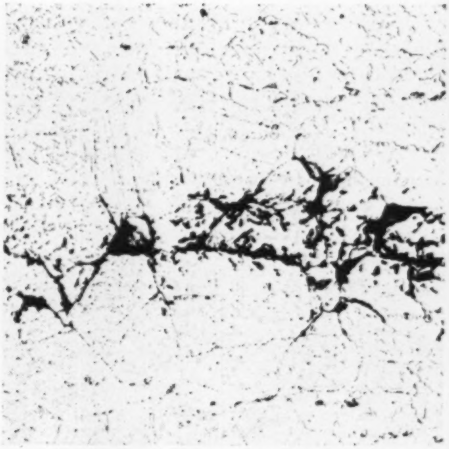


FIG. 12.—Steel No. 2—1500 F—500 hr. Corrosion Rate, 0.00757 ipm (× 500). Reduced to ⅔ size in reproduction.

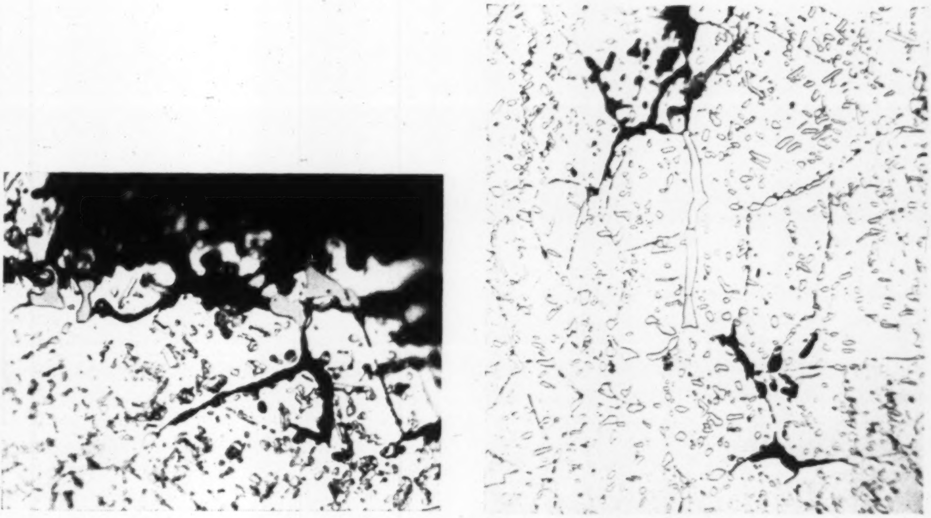


FIG. 13.—Steel No. 2—1500 F—500 hr. Corrosion Rate, 0.00757 ipm. (× 1000). Reduced to ⅔ size in reproduction.

that this constituent is not attacked by nitric acid. As may be seen in Fig. 11, large pools of the constituent exposed to nitric acid showed no sign of attack, nor was there evidence of selective attack on the matrix material immediately adjacent to this constituent.

The 1500 F - 500 hr steel No. 2 sample suffered both intergranular attack and general attack. Metallographic examination of this sample revealed the presence of a large amount of sigma or chi or both. X-ray diffraction confirmed the presence of sigma and chi and indicated that the former was present in only trace amounts. As is illustrated by the photomicrographs (Figs. 12 and 13), the identifiable particles of sigma or chi are not attacked by nitric acid, and there is strong evidence that the matrix material immediately adjoining the sigma or chi particles is at least as resistant as matrix material remote from these particles.

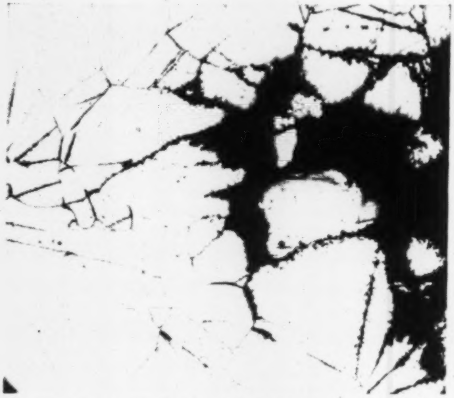


FIG. 14.—Steel No. 3—1350 F—500 hr. Corrosion Rate, 0.147 ipm (× 100). Reduced to $^2/_3$ size in reproduction.

The pattern of general attack that is evident in Fig. 12 is similar to the pattern of occurrence within the grains of sigma or chi, and from this observation one might conclude that these phases, or the matrix material surrounding them, suffer preferential attack. However, if this were true, then particles of sigma or chi that are partially dislodged or partially eaten out should be easy to find, but this is not the case.

Steel No. 3:

By X-ray diffraction a trace of sigma phase was detected in the 1350 F - 500 hr steel No. 3 sample, but this phase could not be identified in the microstructure. As illustrated in Fig. 14, attack on this sample was predominantly intergranular with general attack being moderate. In the samples held at 1500 F for 25 hr, X-ray diffraction results as regards sigma were doubtful, and the phase was not detected metallographically. Corrosion was mainly intergranular as shown in Fig. 15.

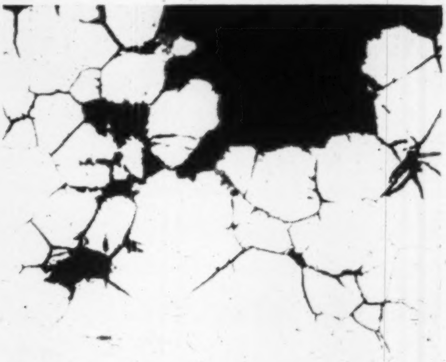
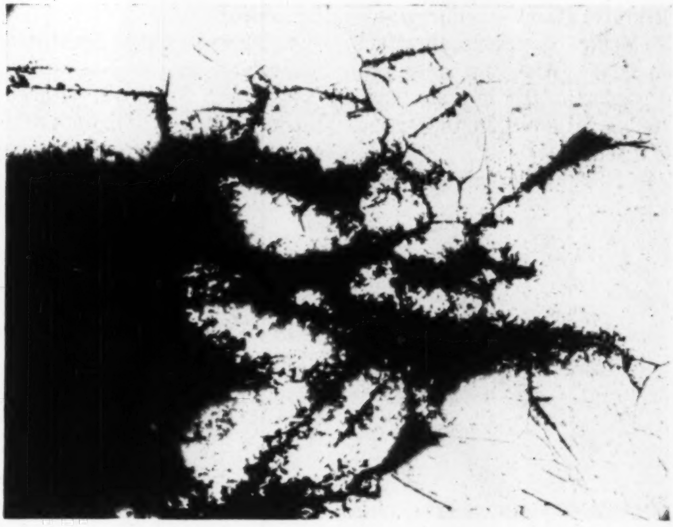


FIG. 15.—Steel No. 3—1500 F—25 hr. Corrosion Rate, 0.0454 ipm (× 100). Reduced to $^2/_3$ size in reproduction.

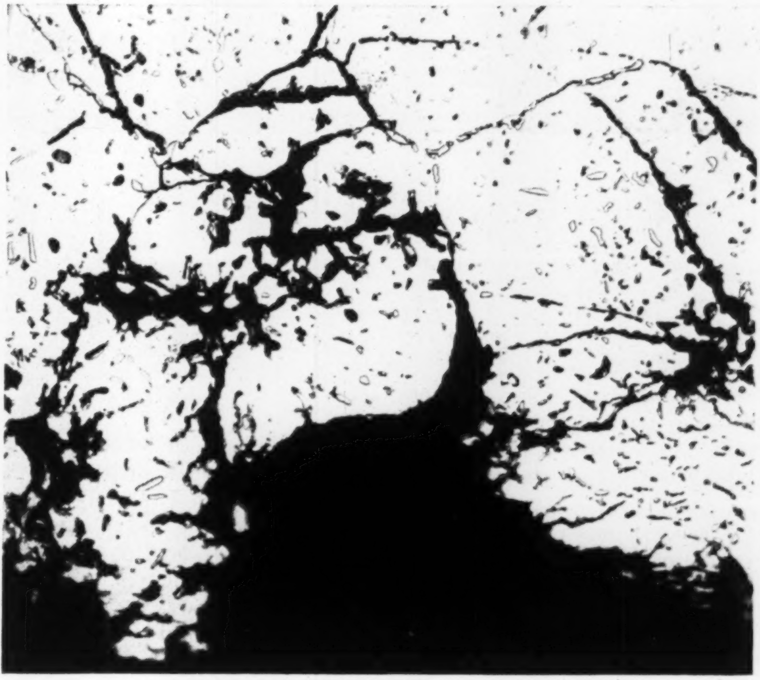
In the case of the 1500 F - 500 hr sample, general attack was heavy although intergranular attack also occurred (Fig. 16(*a*)). The pattern of general attack evident in this specimen (Figs. 16 and 17) is similar to the pattern of occurrence within the grains of sigma or chi. However, this specimen, in common with the sample of steel No. 2 given the same treatment, showed little evidence that nitric acid tends to preferentially attack or dislodge sigma or chi phases. On the contrary, Figs. 16 (*b*) and 17 show a number of places where sigma or chi particles blocked the path of nitric acid attack.

SUMMARY

The findings of this metallographic study may be summarized as follows:



(*a*) (× 100).



(*b*) (× 500).

FIG. 16.—Steel No. 3—1500 F—500 hr. Corrosion Rate, 0.0904 ipm.

1. In the case of all reheated samples of steel No. 1, nitric acid attack was intergranular with no evidence of significant attack upon the grain faces. (With this steel, precipitation was predominantly in grain boundaries, there being little within the grains. No sigma was detected.)

2. With steels Nos. 2 and 3 all specimens showed general attack as well as intergranular attack. (These steels showed pronounced precipitation within the grains after reheating, as well as grain boundary precipitation.)

3. The presence of sigma or chi or both, as relatively large particles in the former ferrite sites of steel No. 2 (specimens heated 500 hr at 1350 and 1500 F), did not result in selective attack upon these sites. It is not possible to prove whether or not sigma phase is a cause of the selective attack on the former ferrite sites of the 1200 F - 500 hr sample.

4. Sigma or chi or both particles that are identifiable in the microstructure of steel Nos. 2 and 3 are not attacked by boiling nitric acid, nor is there evidence that the matrix material immediately adjoining these particles is selectively attacked.

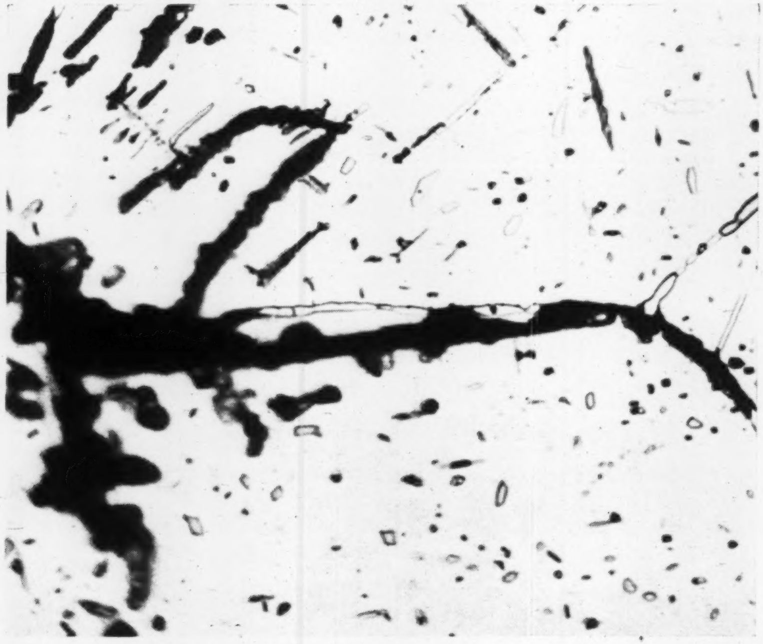


FIG. 17.—Steel No. 3—1500 F—500 hr. Corrosion Rate, 0.0904 ipm (× 1000).

CONCLUSION

There is strong evidence that the relatively high rates of nitric acid attack upon the samples of steels Nos. 2 and 3 heated at 1500 F are not due to the solution of sigma or chi particles nor to accelerated attack upon the matrix material immediately surrounding these particles. Whether or not the over-all depletion of the matrix material in chromium due to sigma and chi formation is a factor, it is not possible to say.

DISCUSSION

Mr. Adolph J. Lena.[1]—The report states: "There is reason to believe that the permeability of steel No. 2 should increase after exposure to 1200 F for 25 hr as it does in steel No. 1. That it tends rather to decrease suggests the formation of some nonmagnetic sigma even at this low temperature although X-ray diffraction tests generally failed to disclose its presence." This suggests that the decrease in magnetism of steel No. 2 after 25 hr at 1200 F is an indication of the presence of sigma.

A word of caution should be inserted with respect to the use of magnetic measurements alone as a means of detecting the transformation of delta ferrite to sigma. Unless the sigma is actually observed either metallographically or by X-ray analysis, the assumption must be made that only the transformation of delta ferrite to sigma can result in a decrease in magnetism. This is not the case, for we have observed that the delta ferrite in a chromium-manganese austenitic steel transforms in a few hours at either 1200 or 1400 F to a lamellar chromium carbide and austenite structure with a complete loss in magnetism. An example of this is shown in Fig. 18. Nehrenberg and Lillys[2] have found a similar transformation in Type 329 steel as well as ferritic stainless steels and tungsten high speed steels. This may be a general type of transformation of the initial decomposition of delta ferrite in other steels as well, and the decrease in magnetism associated with it could lead to erroneous conclusions in regard to sigma formation. That this type of transformation may be operative in the case of the 18-8 Mo steels is indicated by micrographs D-5 and D-9 (Plate D). Micrograph D-5 in particular shows a lamellar-like structure in what must have been prior delta ferrite areas, and the dark etching constituent in this structure has the etching characteristics of carbide rather than sigma. It is attacked and has a black appearance as do the carbides in micrograph D-1 rather than being in relief and light etching as the sigma in micrograph D-9. Our work with the chromium-manganese steels has shown that when this transformation goes to completion, the presence of initial delta ferrite has no accelerating effect on the rate of sigma formation.

It is unfortunate that the corrosion tests were not performed on a low carbon Type 316, for it is not possible to separate the effects of carbide precipitation and sigma formation in higher carbon steels such as those used in this study. It has been found[3] that low carbon steels which are sensitized by sigma formation are dissolved in nitric acid but are immune to attack in the Strauss test. It was also shown that samples which were susceptible after one hour at 1400 F were immune after only 100 hr at 1400 F due to coalescence of the sigma. This effect could not be predicted from the corrosion data in Fig. 6. It might be desirable to emphasize the fact that the data in Fig. 6 represent the combined effect of carbide precipitation and sigma formation, for some might assume that it is due entirely to sigma since no mention is made of chromium carbide in the paper.

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[1] Research Laboratory, Allegheny Ludlum Steel Corp., Pittsburgh, Pa.

[2] A. E. Nehrenberg and Peter Lillys, "High Temperature Transformations in Ferritic Stainless Steels Containing 17 to 25 per cent Chromium," *Preprint No. 31*, Am. Soc. Metals (1953).

[3] A. J. Lena, discussion of unpublished work of J. E. Truman and C. Edeleanu, *Transactions*, Am. Soc. Metals, Vol. 45, p. 466 (1953).

In regard to the title of the report, it would be more appropriate if it referred specifically to chromium-nickel-molybdenum steels rather than chromium-nickel stainless steels in general. Dulis and Smith[4] have shown that the effect of etching reagents varies from one type of steel to another. For instance, electrolytic sodium cyanide rapidly attacks sigma in Types 314, 347, 310, and 321 while it leaves the sigma in 18-8 Mo steels unattacked and in relief. Therefore, the etching techniques described in this paper, at least in some cases, would apply only to the 18-8 Mo steels and not to all austenitic chromium-nickel steels.

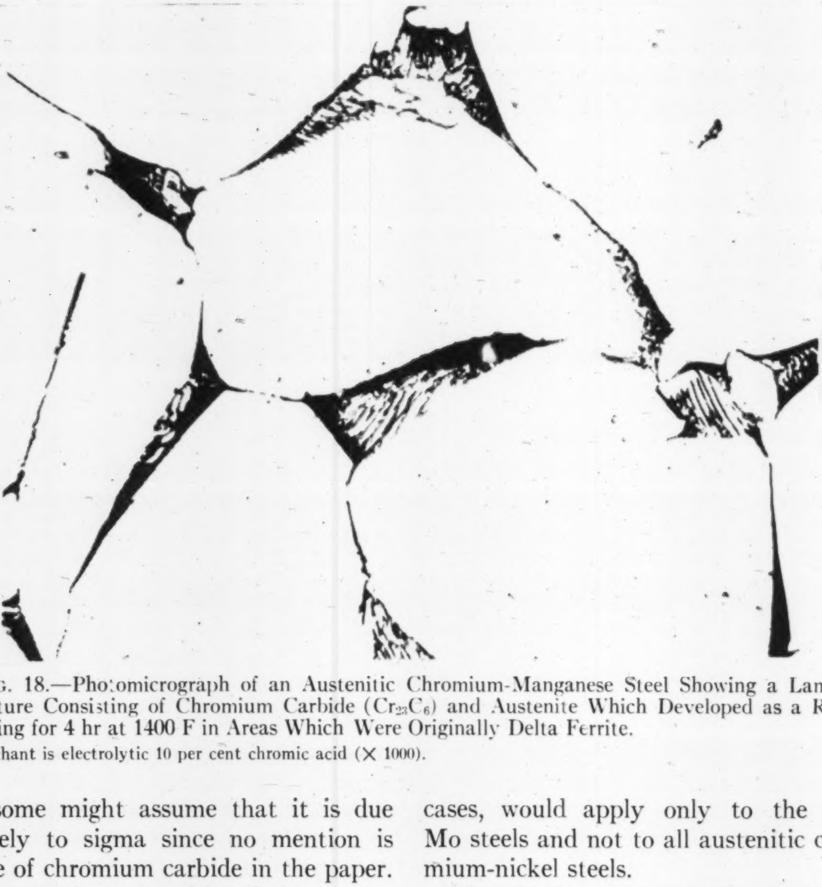

FIG. 18.—Photomicrograph of an Austenitic Chromium-Manganese Steel Showing a Lamellar Structure Consisting of Chromium Carbide ($Cr_{23}C_6$) and Austenite Which Developed as a Result of Aging for 4 hr at 1400 F in Areas Which Were Originally Delta Ferrite.

Etchant is electrolytic 10 per cent chromic acid (× 1000).

MR. T. V. SIMPKINSON.[5]—It might have been well to have pointed out in the report that the term "sigma" is often used in discussing effects which may be due to sigma alone, to sigma plus chi, or even to chi alone. The reader should keep this use of the term in mind.

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[4] E. J. Dulis and G. V. Smith, "Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Chromium-Nickel Steels," Symposium on Nature, Occurrence, and Effects of Sigma Phase, Am. Soc. Testing Mats., p. 3 (1950). (Issued as separate publication *ASTM STP No. 110.*)

[5] Division of Physical Metallurgy, Mines Branch, Department of Mines and Technical Surveys, Ottaw Ont., Can.

I think that further remarks concerning the metallographic work are worth while by way of emphasis. The results of the metallographic examinations made during this investigation show that the presence of sigma or chi phases *can be* proven with reasonable certainty, by metallographic means alone, when the particles of the phase or phases are large enough to be resolved into circles by the light microscope. It was not found possible to distinguish small ("dot-size") particles of sigma or chi from carbides. Knowledge of the chemical composition and thermal treatment, of course, makes interpretation of the microstructure easier and more positive. "Circle-size" particles of sigma or chi are not revealed by 5 to 10 sec etching in Murakami's reagent at room temperature while carbides are, and thus a means of distinguishing carbides is provided. All phases can be revealed by one of the reagents mentioned in the report as suitable for this purpose.

The metallographic studies have indicated that it may be possible to distinguish "circle-size" particles of sigma and chi from one another by means of suitable etching procedures although this was not proven conclusively. Electrolytic sodium cyanide may tint sigma phase while leaving chi uncolored. At 3 v, 10 per cent ammonium persulfate etches out what is believed to be sigma in 30 sec or less, while what is believed to be chi is unaffected after 1 to 2 min. Hot Murakami's reagent and cold modified Murakami's tend to tint the phases differently. Loss of clarity in the printing procedure makes these differences less apparent in the published photomicrographs.

MR. F. B. FOLEY (*closure*).—The warning given by Mr. Lena concerning too hasty a conclusion that mere loss of magnetism is necessarily an indication of sigma formation is well taken. The decrease in permeability could of course result from a transformation of alpha to gamma as well as from alpha to sigma. However, in the case in point, the preponderance of evidence indicates the latter although the situation does not differ essentially from that described by Nehrenberg and Lillys.[2] They observed the transformation to occur isothermally at 1750 F upon cooling from a higher temperature. According to them the precipitation of the chromium carbide, $Cr_{23}C_6$, in lamellar form took sufficient chromium from the delta iron to cause it to transform to austenite which, upon cooling, transformed to martensite. The case in point involves transformation occurring in steel No. 2 during reheating, not isothermally during cooling; nevertheless the conditions are similar, involving the precipitation of a $Cr_{23}C_6$ type carbide out of ferrite in a molybdenum-bearing 18-8 alloy. The carbide is probably not so rich in chromium, which may be replaced by molybdenum to give $(MoCr)_{23}C_6$. In the Nehrenberg-Lillys steel the austenite transforms to martensite upon cooling. Were this to occur in steel No. 2, permeability would not decrease and might increase as it does in steel No. 1. The phase which forms at 1200 F and which reduces permeability does not transform during cooling and reduces the impact value. It is not to be expected that an increase in the amount of austenite would lessen this value. Furthermore, Nehrenberg and Lillys apparently relied entirely on metallographic appearance in their identification of the martensite and then, by deduction, reasoned that it formed from austenite. Acknowledging that their reasoning appears sound, it would be entirely convincing had they proved their point by other means such as by X-ray diffraction and magnetic analysis. In steel No. 2 it is quite evident that the amount of material involved in the transformation is too small for identification by metallographic means or convincingly by X-ray diffraction. Laboratory No. 3 reported "inconclusive" evidence of sigma after 25 hr at 1200 F and its presence definitely after 500 hr; laboratories Nos. 1 and 4 reported a trace of both sigma and chi after 500 hr exposure; and other laboratories reported no indication after the 1200 F reheatings.

Mr. Simpkinson's comments on metallographic detection of sigma are very pertinent. The identification of sigma, metallographically, involves all the difficulties inherent in the study of any constituent under the microscope, requiring, as it does, a prior knowledge of the composition and thermal history of the specimen together with a pretty thorough knowledge of the equilibrium phase diagram of the alloy system involved. There is a particle size limitation in identification by both microscopical and X-ray diffraction methods, and evidence persists that sigma, beyond the range of detection by either instrument, can profoundly affect the mechanical, magnetic, and chemical properties of the metal.

The dark-etching lamellar-like constituent in micrographs D-5 and D-9, to which Mr. Lena refers, is carbide and the gray areas in which it occurs are sigma which now occupies the site of the delta iron from which it formed—there is no evidence here of the lamellar carbide-austenite structure described by Nehrenberg and Lillys.[2] The lamellar carbide-sigma eutectoidal type structure is not uncommon in sigma-forming alloys.

As Mr. Lena points out, much of the attack shown in Table IX and in Fig. 6 has resulted from the combined effect of carbide and sigma formation, and it is difficult to separate the two effects. No conclusions with regard to the effect of the two phases were drawn and Mr. Lena's remarks are therefore very welcome indeed. Sigma, like chromium carbide, is presumed to take a large amount of chromium from the adjacent metal, and in this respect its effect on corrosion resistance would be expected to be rather like that associated with the formation of carbides. Shirley[6] concluded that sigma itself is highly resistant to attack by nitric acid but found that its formation led to considerably increased attack on adjacent metal. Work on carbonless or extra low carbon 18-8 plus molybdenum might enable differentiation between attack due to carbides and that due to sigma formation.

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[6] H. T. Shirley, "Microstructural Characteristics of Acid Corrosion in 18 per cent Cr, 8–14 per cent Ni, 3 per cent Mo Steel," *Journal*, Iron and Steel Inst., July, 1953, p. 242

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: one in Washington, D. C., on October 10, 1952; one in New York, N. Y., on January 9, 1953; and one in Cleveland, Ohio, on April 21, 1953. Meetings of Subcommittees II, IV, and VII also were held during the year.

The committee now consists of 76 members, of whom 36 are classified as producers, 27 as consumers, and 13 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee B-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (B 172 – 51 T),

Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members for Electrical Conductors (B 173 – 51 T), and

Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 51 T).

These recommendations were accepted by the Standards Committee on August 1, 1952, and the revised specifications appear in the 1952 Book of ASTM Standards, Part 2.

On March 10, 1953, the Administrative Committee on Standards accepted the Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors submitted by Committee B-1. The method will appear in the 1953 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 263 – 53 T.

NEW TENTATIVES

For many years there has been need for industry standardization on stiffness requirements for soft copper wire, particularly as applied to magnet wire, as evidenced by the development of many special test methods by individual users (Morehouse, RCA, Belden, LSE, Rockwell Superficial, etc.). The committee has considered this problem over the years but has been unable to establish one method as being applicable for all requirements. Therefore, in order to provide a start in this direction, it has limited its present consideration to a narrower scope covering only bare soft square and rectangular copper wire. The committee, therefore, recommends for publication as tentative the proposed Method of Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication, as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends that the following nine specifications be revised as indicated and continued as tentative:

Tentative Specifications for Hard-Drawn Copper Wire (**B 1 – 52 T**).[2]—Revise as follows:

Section 4(a).—Change the parenthetical insert "(Note 1)" to read "(Notes 1 and 2)."

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 2.

[2] 1952 Book of ASTM Standards, Part 2.

Table I.—In the heading of the fourth column, delete the reference to footnote *c* and insert after "psi" the words "(Note 2)."

In the footnotes, delete footnote *c*.

Explanatory Notes.—Renumber Note 2 to read Note 3 and renumber all subsequent notes accordingly. Insert a new Note 2, to read as follows:

> NOTE 2.—The tensile strength values given in Table I cannot always be met if the wire is drawn into coils of less than 19 in. inside diameter or if the wire has been rewound. Lower tensile strength values, therefore, are permissible for smaller diameter coils, or rewound wire, through mutual agreement between the manufacturer and the purchaser at the time the order is placed.

To conform with this change, renumber the note references in footnote *a* of Table I, and in Sections 4(*b*), 4(*d*), 5(*a*), 5(*b*), 5(*c*), 6(*a*), 6(*b*), 7 (heading), and 8.

Tentative Specifications for Soft or Annealed Copper Wire (**B 3 - 52 T**).[2]—The specifications in their proposed revised form are appended hereto.[3]

Tentative Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (**B 33 - 52 T**).[2]—Revise as follows:

Section 1.—Delete the word "suitable."

Section 3.—Revise Paragraph (*a*) to read "*Tin for Coating.*—The tin used for coating shall be commercially pure (Note 1)."

Revise the first sentence of Paragraph (*b*) to read "*Copper-Base Metal.*—The copper in the wire shall conform to the requirements of the standard specifications of the American Society for Testing Materials, as follows:"

Retain the remaining sentences of Paragraph (*b*) without change.

Section 4.—Revise Paragraph (*b*) to read "Wire whose nominal diameter is more than 0.001 in. (1 mil) greater than a size listed in Table I, but less than the next larger size, shall conform to the requirements of the next larger size."

Revise Paragraph (*d*) to read "*Retests.*—If upon testing a specimen from any coil or spool of wire, the results do not conform to the requirements prescribed in Table I, two additional specimens shall be tested, and the average of the three tests shall be taken as the value for the coil or spool."

Section 5.—In the first sentence, change the word "Electric" to read "Electrical."

In the second sentence, insert the words "of tinned wire" between the words "resistivity" and "shall."

Section 6.—In Paragraph (*b*), insert the word "tinned" between the words "The" and "wire."

Revise Paragraph (*c*) to read as follows:

> (*c*) The diameter of tinned wire in coils shall be measured at three places, one near each end and one near the middle of the coil. In the case of spools, approximately 12 ft shall be unreeled and the diameter of the tinned wire shall be measured in six places between the second and twelfth foot from one end. The average of the measurements obtained on any coil or spool shall be within the limits specified in Paragraph (*b*).

Section 7.—Revise Paragraph (*a*) to read "The coating on the wire shall be continuous."

Revise Paragraph (*b*) to read as follows:

> (*b*) The continuity of coating on the wire shall be determined on representative samples taken before stranding or insulating. The continuity of tinning shall be determined by the hydrochloric acid - sodium polysulfide test in accordance with Sections 8 to 10.

Section 8(*b*).—Revise the second sentence to read "The specimens thus cleaned shall be kept wrapped in a clean, dry cloth until required for test."

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[3] The revised specifications were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 2.

Section 13(b).—Change the word "from" to read "of."

Section 15(b).—Insert the word "tinned" between the words "The" and "wire."

Section 17.—Revise to read "Wire that fails to conform to the requirements of these specifications shall be rejected."

Explanatory Notes.—In Note 1, delete the first two sentences, and replace with a new opening sentence, reading "It is necessary that the coating of tin on the wire be continuous."

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (**B 172 - 52 T**).[2]—Revise as follows:

Section 11(c).—Revise to read as follows:

> (*c*) The area of cross-section of a conductor shall be determined by the Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors (ASTM Designation: B 263).[4] In applying this method, the increment of weight resulting from stranding may be the applicable value listed in Note 8, or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

Explanatory Notes.—In Note 8, first sentence, delete the words "of increase." In the heading of the right-hand column of the tabulation, delete the words "of Increase."

Tentative Specifications for Rope-Lay Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (**B 173 - 52 T**).[2]—Revise as follows:

Section 11(c).—Revise to read as follows:

> (*c*) The area of cross-section of a conductor shall be determined by the Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors (ASTM Designation: B 263).[4] In applying this method, the increment of weight resulting from stranding may be the applicable value listed in Note 7, or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

Explanatory Notes.—In Note 7, first sentence, delete the words "of increase." In the heading of the right-hand column of the tabulation, delete the words "of Increase."

In Note 8, second sentence, delete the words "of increase."

Tentative Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors (**B 174 - 52 T**).[2]—Revise as follows.

Section 10(a).—In the second sentence delete the words "of increase."

Section 11(c).—Revise to read as follows:

> (*c*) The area of cross-section of a conductor shall be determined by the Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors (ASTM Designation: B 263).[4] In applying this method, the increment of weight resulting from stranding may be the applicable value specified in Section 10(*a*), or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

Tentative Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (**B 189 - 52 T**).[2]—Revise as follows:

Section 1.—Insert the words "or annealed" between the words "soft" and "copper."

Section 10.—Revise to read "Necessary joints in the completed wire and in the wire and rods prior to final drawing shall be made in accordance with the best commercial practice."

Explanatory Notes.—At the end of Note 3, add a new sentence to read "Relationships which may be useful in connection with the values of resistivity prescribed in these specifications are as shown in Table IV, each column containing equivalent expressions at 20 C (68 F)."

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[4] 1953 Supplement to Book of ASTM Standards, Part 2.

New Table.—Add a new Table IV on resistivity values to read the same as the accompanying Table I.

Tentative Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (**B 230 - 52 T**).[2]—Revise as follows:

Section 4.—Change the reference to Sections 5 and 6 to read "... of Sections 5 and 7."

New Section.—Renumber Section 6 to be Section 7 and renumber all following sections accordingly. Insert a new Section 6 to read as follows:

6. *Bending Properties* (*a*) The wire shall be free from brittleness as evidenced by its ability to be coiled or looped around its own diameter either with or without a mandrel. No fracture shall occur. Slight surface checks shall not constitute cause for rejection.

(*b*) Any coil or reel may be tested in accordance with Paragraph (*a*), but the frequency of production sampling and testing shall be by agreement between the manufacturer and the purchaser.

Explanatory Note 2.—In the second paragraph, change "Section 6" to read "Section 7."

Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (*ACSR*) (**B 232 - 52 T**).[2] —Revise as follows:

TABLE I.—RESISTIVITY VALUES.

Conductivity at 20 C (68 F), per cent	100.00	97.66	97.16	96.16	94.16	93.15
Ohms (mile, lb)	875.20	896.15	900.77	910.15	929.52	939.51
Ohms (meter, g)	0.15328	0.15694	0.15775	0.15940	0.16279	0.16454
Ohms (mil, ft)	10.371	10.619	10.674	10.785	11.015	11.133
Ohms (meter, sq mm)	0.017241	0.017654	0.017745	0.017930	0.018312	0.018508
Microhms—in	0.67879	0.69504	0.69863	0.70590	0.72092	0.72867
Microhms—cm	1.7241	1.7654	1.7745	1.7930	1.8312	1.8508

Section 4.—Reletter the present paragraph to read "Paragraph (*b*)" and reinsert Paragraph (*a*) (from B 232 - 50 T) to read as follows:

(*a*) Electric butt welds in the finished individual aluminum wires composing the conductor may be made during the stranding process. No weld shall occur within 50 ft of a weld in the same wire or in any other wire of the completed conductor.

Section 7.—Reletter Paragraph (*b*) to Paragraph (*c*) and insert a new Paragraph (*b*) to read as follows:

(*b*) All aluminum wires composing the conductors shall be capable of meeting the bending properties stated in the Tentative Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (ASTM Designation: B 230)[4] after stranding. Routine production testing after stranding is not required.

Section 11.—Revise to read as follows:

11. *Variation in Area.*—The area of cross-section of the aluminum wires of a conductor shall not be less than 98 per cent of the area specified. Unless otherwise specified by the purchaser, the manufacturer may have the option of determining the cross-sectional area by either of the following methods, except that in case of question regarding area compliance, Method (*2*) shall be used:

(*1*) The area of cross-section may be determined by calculations from diameter measurements, expressed to four decimal places, of the component aluminum wires at any point when measured perpendicularly to their axes.

(*2*) The area of cross-section of the aluminum wires of a conductor may be determined by the Tentative Method for Determination of Cross-Sectional Areas of Stranded Conductors (ASTM

Designation: B 263).[4] In applying that method, the increment in weight resulting from stranding may be the applicable value specified in Section 10(*a*) or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in the following four standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

TABLE II.—TENSILE REQUIREMENTS FOR GROOVED AND FIGURE-9 TROLLEY WIRE.

Nominal Area, cir mils	Alloys 40 and 55 Bronze Wire		Alloy 65 Bronze Wire		Alloy 80 Bronze Wire	
	Tensile Strength, min, psi	Elongation in 10 in., min, per cent	Tensile Strength, min, psi	Elongation in 10 in., min, per cent	Tensile Strength, min, psi	Elongation in 10 in., min, per cent
Grooved:						
350 000	62 500	4.00	56 200	4.00	59 500	4.00
300 000	64 800	4.00	57 800	4.00	61 500	4.00
211 600	69 000	3.25	61 000	3.25	65 000	3.25
168 100	71 000	2.75	63 000	2.75	67 000	2.75
133 200	73 000	2.25	65 000	2.25	69 000	2.25
Figure-9:						
335 000	61 500	4.00	54 000	4.00	56 800	4.00

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (**B 8 - 52**).[2]—Revise as follows:

Section 10(a).—In the second sentence delete the words "of increase."

Table III.—In the heading of the right-hand column, delete the words "of Increase."

Section 11.—Retain the present Section 11 as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) The area of cross-section of a conductor may be determined by the Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors, (ASTM Designation: B 263).[4] In applying this method, the increment of weight resulting from stranding may be the applicable value specified in Section 10(*a*), or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

Standard Specifications for Bronze Trolley Wire (**B 9 - 52**).[2]—Revise as follows:

Section 1.—Revise to read as follows:

1.(*a*) These specifications cover round, grooved, and figure-9 deep-section grooved bronze trolley wire.

(*b*) The bronze trolley wire may be made in any of the four distinct alloys indicated below in accordance with their increasing conductivities.

Alloy 40
Alloy 55
Alloy 65
Alloy 80

Section 2.—Revise Item (*4*) to read "Alloy (Section 1(*b*))."

Section 3.—In Paragraph (*a*), change the parenthetical insert "(Note 2)" to read "(Note 1)."

Delete Paragraph (*b*) including footnote 3.

Table I.—Change column headings, as follows:

"Class A Bronze Wire" to read "Alloys 40 and 55."

"Class B Bronze Wire" to read "Alloy 65."

"Class C Bronze Wire" to read "Alloy 80."

Section 4(c).—Change parenthetical insert "(Note 3)" to read "(Note 2)."

Section 7.—Change the heading preceding Section 7 to read "Grooved and Figure-9 Wire." Change Paragraph (*a*) to read "Grooved and figure-9 wire shall conform to the applicable requirements as to tensile properties prescribed in Table III."

In Paragraph (*b*), insert the words "or figure-9" between the words "grooved" and "wire."

In Paragraph (*c*), insert the words "or figure-9" between the words "grooved" and "wire."

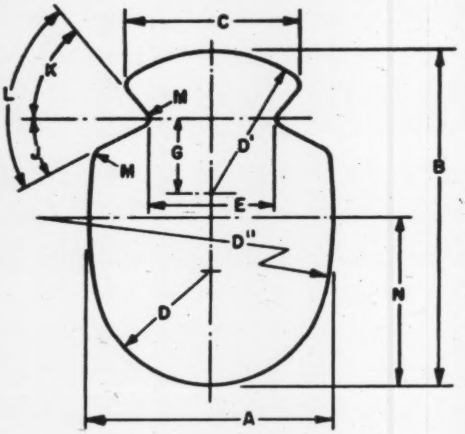
Table III.—Revise to read as shown in the accompanying Table II.

Section 8.—Add at the end of the present section, another sentence reading "The twist test shall be omitted for figure-9 wire."

Section 9.—Retain the present Section 9 as Paragraph (*a*) and add a new Paragraph (*b*) to read "The standard section of figure-9 wire shall be as shown in Fig. 2 (accompanying Fig. 1)."

New Figure.—Insert a new Fig. 2, as shown in the accompanying Fig. 1.

Section 10.—Change Paragraph (*a*) to read "The size of the trolley wire shall be expressed as the nominal area of cross-section in circular mils."



Nominal Size, cir mils	335 000
Area, sq in (Note 7)	0.2642
Area, cir mils (Note 7)	336 400
Weight, lb per Mile (Note 7)	5386
DIMENSIONS FOR INSPECTION, IN.	
A	0.482 $\begin{cases}+0.006\\-0.012\end{cases}$
B	0.680 ± 0.012
DIMENSIONS FOR REFERENCE, IN.	
C	0.376 ± 0.007
D—Radius	0.235
D′—Radius	0.275
D″—Radius	0.960
E	0.267 $\begin{cases}+0.005\\-0.010\end{cases}$
G	0.093
J	27 ± 2 deg
K	51 ± 2 deg
L	78 deg
M—Radius	0.015 $\begin{cases}+0.010\\-0.005\end{cases}$
N	0.342

FIG. 1.—Standard Section of Figure-9 Grooved Trolley Wire.

Insert new Paragraphs (*b*) and (*c*) to read as follows:

> (*b*) The standard sizes of grooved trolley wire shall be as specified in Fig. 1.
>
> (*c*) The standard size of figure-9 wire shall be as specified in Fig. 2 (accompanying Fig. 1).

Reletter the present Paragraph (*b*) to Paragraph (*d*), insert the words "and Figure-9" between the words "grooved" and "trolley," and add at the end the words "for grooved wire, and Fig. 2 for figure-9 wire."

Reletter the present Paragraph (*c*) to Paragraph (*e*) and change "(Note 7)" to read "(Note 6)."

Section 11.—Change the heading preceding Section 11 to read as follows: "Round, Grooved, and Figure-9 Wire."

In Paragraph (*a*), change the word "Electric" to read "Electrical," and change "(Note 4)" to read "(Note 3)."

Table IV.—Revise to read as shown in the accompanying Table III, deleting Footnote *a*.

Section 12.—Change "(Note 5)" to read "(Note 4)," and change "(Note 6)" to read "(Note 5)."

Figure 1.—Change "(Note 8)" to read "(Note 7)" in three places.

Explanatory Notes.—Delete the present Note 1 and renumber successive notes accordingly.

Renumber Note 7 to read Note 6, and insert in the second sentence after the words "Fig. 1" the words "for grooved wire, and Fig. 2 for figure-9 wire."

TABLE III.—REQUIREMENTS FOR ELECTRICAL RESISTIVITY.

Wire	Resistivity at 20 C (68 F) ohms (mile, lb)
Alloy 40	2188
Alloy 55	1591
Alloy 65	1346
Alloy 80	1094

Renumber Note 8 to read Note 7, and add after the words "Fig. 1," the words "for grooved wire and Fig. 2 for figure-9 wire."

Table V.—Insert a new column of figures between the conductivities 65 and 40, as follows:

Conductivity at 20 C (68 F), per cent	55.00
Ohms (mile, pound)	1591.27[a]
Ohms (meter, gram)	0.27868
Ohms (mil, foot)	18.857
Ohms (meter, square millimeter)	0.031348
Microhms-inch	1.2342
Microhms-centimeter	3.1348

Revise Footnote *a* to read "These values are carried out to two decimal places to indicate somewhat more precisely than in Section 11(*a*) the resistivity equivalent to 55 and 65 per cent conductivity."

Editorial Note.—In the present proposed Section 10(*c*), renumber to read Section 10(*e*), and in the first sentence, after the words "Fig. 1" insert the words "and Fig. 2."

In the section titled "New Figure," change the words "Fig. 2" to read "Fig. 3."

In the section titled "Note 7," change to "Note 6," and revise to read "Delete Note 6 and renumber Note 7 as Note 6."

Renumber the proposed Fig. 2 to read Fig. 3. In the tabulation in this same Figure, insert in the heading of the last 2 columns the figures "335,000" between the figures "300,000" and "350,000."

Standard Specifications for Hard-Drawn Copper Alloy Wires for Electrical Conductors (**B 105 - 52**).[2]—Revise as follows:

Section 1(b).—Revise to read as follows:

> (*b*) The copper alloy wires may be made in any one of ten distinct alloys designated 8.5 to 85 in accordance with their increasing conductivities, as follows:
>
> Alloy 8.5
> Alloy 13
> Alloy 15
> Alloy 20
> Alloy 30
> Alloy 40
> Alloy 55
> Alloy 65
> Alloy 80
> Alloy 85

Section 2.—Revise Item (*3*) to read "Alloy (Section 1(*b*) and Table I)."

Section 3(b).—Delete Paragraph (*b*) including Footnote 3.

Section 4(b).—Change the word "grades" to read "alloys," in two places.

Table I.—In the column headings, change the words "Grade No." or "Grade Nos." to read "Alloy" or "Alloys," respectively.

Section 7.—In the first sentence, change "Electric" to read "Electrical."

In the second sentence, change the word "grade" to read "alloy."

In the tabulation, change the heading of the first column to read "alloy" and delete the words "No." in all cases.

Section 9.—In the tabulation, change the heading of the first column to read "Alloy" and delete the words "No." or "Nos." in all cases.

Explanatory Notes.—In Note 1, change the word "grades" to read "alloys" in two places.

In the tabulation, change the heading of the first column to read "Alloy" and delete the words "No." in all cases.

Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn (**B 231 - 52**).[2]—Revise as follows:

Section 7.—Add a new Paragraph (*d*) to read as follows:

(*d*) All wires composing the conductors shall be capable of meeting the bending properties stated in the Tentative Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (ASTM Designation: B 230)[2] after stranding. Routine production testing after stranding is not required.

Section 10.—Revise to read as follows:

10. *Variation in Area.*—The area of cross-section of the completed conductor shall not be less than 98 per cent of the area specified. Unless otherwise specified by the purchaser, the manufacturer may have the option of determining the cross-sectional area by either of the following methods, except that in case of question regarding area compliance, Method (*2*) shall be used.

(*1*) The area of cross-section of a conductor may be determined by calculations from diameter measurements, expressed to four decimal places, of its component wires at any point when measured perpendicularly to their axes.

(*2*) The area of cross-section of a conductor may be determined by the Tentative Method for Determination of Cross-Sectional Areas of Stranded Conductors (ASTM Designation: B 263).[4] In applying that method, the increment in weight resulting from stranding may be the applicable value specified in Section 9(*a*) or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEE

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of the various specifications.

Subcommittee II on Methods of Test and Sampling Procedures (J. B. Dixon, chairman) completed preparation of the proposed revision of Specification B 3 to incorporate statistical sampling procedures, completed its review of the proposed Tentative Method for Determination of Cross-Sectional Area of Stranded Conductors, and is considering the desirability of ultimately developing statistical sampling procedures for tinned-copper, alloy-coated copper, and hard-drawn aluminum wire.

Subcommittee IV on Conductors of Copper and Copper Alloys (B. J. Sirois, chairman) completed preparation of the proposed Tentative Method of Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication, and revisions of specifications to cover the addition of a 55 per cent conductivity alloy for bronze trolley wire, and the addition of a figure-9, deep-section bronze trolley wire.

Task groups are working on the preparation of proposed Tentative Specifications for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment, and are considering the need for specifications to cover silver-plated wire or other plated or coated wires for higher-temperature applications.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VII on Conductors of Light Metals (P. V. Faragher, chairman) has completed development of a ductility (brittleness) test requirement for hard-drawn aluminum wires, is working on the standardization of direction of lay of stranded aluminum conductors, standardization of large sizes of stranded aluminum conductors, standardization of additional constructions of ACSR conductors, preparation of a proposed specification for annealed or one-quarter hard aluminum wire for telephone cable applications, and is considering the development of specifications for aluminum wire to be used in magnet wire.

This report has been submitted to letter ballot of the committee, which consists of 76 members; 56½ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys has held no meeting since its meeting in New York, N. Y., on June 26, 1952. During the past year the work of the committee has been handled by correspondence.

The following six subcommittees have work before their respective task groups, namely, Subcommittee I on Refined Copper; Sub-committee II on Refined Lead, Tin, Antimony and Bismuth; Subcommittee III on White Metal Alloys; Subcommittee VI on Coated Metals; Subcommittee VII on Refined Nickel and Cobalt, High Nickel Alloys and High Cobalt Alloys, Cast and Wrought; and Subcommittee VIII on Miscellaneous Refined Metals and Alloys. At present the other subcommittees have no actual work before them although work is pending.

The Advisory Committee met on June 25, 1952 and again on February 3, 1953, both meetings being held in New York, N. Y. At this time the committee has no recommendations in connection with standards or tentatives under its jurisdiction.

A Symposium on Tin was conducted at the 1952 Annual Meeting of the Society by Subcommittee II on Refined Lead, Tin, Antimony and Bismuth. This Symposium as published[1] will comprise the following chapters:

Tin Production and Resources.
The Production and Uses of Tin Coatings.
Trends in the Use of Tin in the Container Industry.
Tin in Automobile Body Construction.
Effect of Impurities in Tin on the Properties and Uses of the Metal and Alloys Containing Tin.
The Determination of Small Amounts of Impurities in Tin.
Panel Discussion on the Analysis of Tin, which was conducted in cooperation with Committees E-2 and E-3.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (William E. Milligan, chairman) has before it the revision of Specifications on Fire-Refined Casting Copper (B 72 – 47 T), and Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 42). The work of revision of each specification is in the hands of respective task groups.

Subcommittee II on Refined Lead, Tin, Antimony and Bismuth (Sidney Rolle, chairman) still has under discussion the matter of a specification for pig tin. There is a variance of opinion among the members with respect to such specifications, and a task group is now looking into the preparation of a classification of the various grades of tin. This task group met in New York, N. Y., on February 4, 1953. It is expected that further meetings of this group, prior to the Annual Meeting of the Society, will make a report available for use of the subcommittee.

Subcommittee III on White Metals and Alloys (G. H. Clamer, chairman).—The draft of a proposed tentative specification for rosin flux cored solder which was sent to letter ballot and failed to receive acceptance has been returned to the task group for further consideration.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Symposium on Tin, issued as separate publication *ASTM STP No. 141.*

Subcommittee VI on Coated Metals (F. L. Scovill, Jr., chairman) continues the study of methods of test for continuity of coating for Standard Specification for Lead-Coated Copper Sheets (B 101 – 40).

Subcommittee VII on Refined Nickel and Cobalt, High-Nickel Alloys and High-Cobalt Alloys, Cast and Wrought (O. B. J. Fraser, chairman) has before it for revision the following tentative specifications:

Nickel-Copper Alloys Plate, Sheet and Strip (B 127 – 49 T),
Nickel Rods and Bars (B 160 – 49 T),
Nickel Seamless Pipe and Tubing (B 161 – 49 T),
Nickel Plate, Sheet and Strip (B 162 – 49 T),
Seamless Nickel and High Nickel Alloy Condenser, Evaporator and Heat-Exchanger Tubes (B 163 – 49 T),
Nickel-Copper Alloy Rods and Bars (B 164 – 49 T),
Nickel-Copper Alloy Seamless Pipe and Tubing (B 165 – 49 T),
Nickel-Chromium-Iron Alloys Rods and Bars (B 166 – 49 T),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tubing (B 167 – 49 T), and
Nickel-Chromium-Iron Alloy Plate, Sheet and Strip (B 168 – 49 T).

The proposed revisions of these tentatives failed to receive acceptance in letter ballot of the subcommittee and were returned to the task group for further revision.

In addition, the subcommittee is preparing tentative specifications for nickel-molybdenum alloy (Hastelloy B), and nickel-chromium-molybdenum alloy (Hastelloy C).

The subcommittee is also working in conjunction with the ASME Boiler Code Committee on specifications for high-nickel alloy castings.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—The proposed Tentative Specifications for Iodide Titanium; Titanium Strip, Sheet, Plate, Bar, Tube, Rod and Wire; and Titanium Ingot have been accepted by the Society. The subcommittee has before a task group a tentative specification for sponge titanium.

At the meeting on June 26, 1952, the committee re-elected Bruce W. Gonser chairman, and G. Howard LeFevre secretary, both to serve for two years.

This report has been submitted to letter ballot of the committee, which consists of 135 members; 107 members returned their ballots, of all whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

G. HOWARD LEFEVRE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards proposed Tentative Specifications for Rosin Flux Cored Solder. This recommendation was accepted by the Standards Committee on November 9, 1953, and the new tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 284 – 53 T.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in New York, N. Y., on June 23, 1952.

The committee consists of 103 members, of whom 85 are voting members; 29 are classified as consumers, 36 as producers, and 20 as general interest members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the three test methods under its jurisdiction be continued as tentative pending possible revisions from work planned:

Tentative Methods of Tests for:

Total Immersion Corrosion Testing of Non-Ferrous Metals (B 185 - 43 T),
Salt Spray Fog Testing (B 117 - 49 T), and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (B 192 - 44 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (C. O. Durbin, chairman).—The task group which has been studying the acetic acid - salt spray test has continued its work and found that this spray test is as effective as the regular salt spray test for determining the quality of copper-nickel-chromium or nickel-chromium coatings on steel. It is also indicated from the work of the task group that in the acetic acid - salt spray test, time to failure of zinc-coated steel is proportional to the thickness of the zinc coating.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—A task group has been appointed to arrange for the determination of changes in weight and tensile properties of all the specimens removed from the atmospheric test racks after about 20 years.

The same task group will arrange for the exposure of a few specimens of several metals at the new ASTM test sites in order to calibrate the corrosivity of the new sites with those of the old sites.

Subcommittee VII on Weather (F. L. LaQue, chairman).—L. G. vonLossberg, who has been making a survey of instruments and methods for measuring atmospheric pollution, particularly with respect to the presence of corrosive gases, presented an interim report to the subcommittee.

The task group consisting of C. P. Larrabee and O. B. Ellis has continued its program of calibrating the corrosivity of the atmosphere at each of the several ASTM test sites by exposing specimens of zinc and steel. An interim report on these tests is to be presented at the Annual Meeting of the Society as a contribution of Subcommittee VII. This report is appended hereto.[1]

Subcommittee VIII on Galvanic and Electrolytic Corrosion (Gerald Kingsley, chairman).—A task group has been appointed to obtain data on the first group of disk couples of magnesium coupled with other metals, removed from the atmospheric exposure test racks.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] See p. 194.

Exposure of the spool type samples of Part II of the magnesium galvanic couple program has been completed, and results are now being evaluated.

Part III of the atmospheric exposure test on magnesium coupled with other metals, consisting of magnesium plates to which are fastened bars of other metals, has been delayed due to loss of materials.

The subcommittee plans to repeat some of the original couple tests made several years ago on a few common metals in order to obtain comparative data on the corrosivity of old and new ASTM test sites.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 52 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

APPENDIX

PROGRESS REPORT OF A SUBGROUP[1] OF SUBCOMMITTEE VII, ON CORROSIVENESS OF VARIOUS ATMOSPHERIC TEST SITES AS MEASURED BY SPECIMENS OF STEEL AND ZINC*

During the February, 1948, meeting of ASTM Committee B-3, Subcommittee VII on Weather, the effects of periodic variation in weather conditions on atmospheric corrosion were discussed. To determine the magnitude of such variations C. P. Larrabee and O. B. Ellis[1] were appointed as a task group to make periodic exposures of specimens of steel and of zinc at several atmospheric test sites. The first exposures were made in April, 1948. Annual exposures and removals have been made since. This is a progress report summarizing some of the data that have been accumulated.

Plan of Test:

The test program was limited to two materials: cold-rolled steel and sheet zinc. Four specimens, 4 by 6 in., of each material were exposed for each removal period at each test site. The steel specimens were identified by notches punched in the edges, and the zinc specimens by drilled holes. The identification code designated the time of exposure and removal and the location of the specimens.

The steel specimens were pickled and weighed before exposure. After exposure they were cleaned in molten sodium hydroxide - sodium hydride and reweighed. The zinc specimens were degreased and weighed before exposure. After removal from test they were cleaned in dilute ammonium hydroxide and reweighed.

The specimens were mounted on open frames between porcelain insulators. The frames were at an angle of 30 deg from the horizontal, facing south.

TABLE I.—SCHEDULE FOR EXPOSURE AND REMOVAL OF ATMOSPHERIC CALIBRATION TESTS.

Exposure	Removal Dates							
	1949	1950	1951	1952	1953	1954	1955	1956
1948	x	x	...	x	...	...	...	x
1949	...	x	x	...	x	...	...	x
1950	...	...	x	x	...	x	...	x
1951	...	...	...	x	x	...	x	...
1952	...	...	...	...	x	x	...	x
1953	...	...	...	...	...	x	x	x
1954	...	...	...	...	...	...	x	x
1955	...	...	...	...	...	...	...	x

Several samples of steel and of zinc, each consisting of four 4 by 6-in. specimens, have been and will be exposed according to the schedule shown in Table I. From this table it is evident that cumulative data for exposure periods greater than one year will be obtained. However, for this discussion only the 1-yr weight-loss data of steel and of zinc specimens exposed at seven locations will be considered.

Presentation of Data:

The 1-yr weight-loss data of steel and

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] C. P. Larrabee, Research and Development Laboratory, United States Steel Corp., Vandergrift, Pa. and O. B. Ellis, Senior Research Engineer, Armco Steel Corp., Middletown, Ohio.

of zinc specimens exposed at seven locations are given in Table II. From this tabulation it can be seen that there were annual variations in weight losses of steel and of zinc at all locations and that the several locations showed differing severity of corrosive attack. However, at two locations the variations in annual weight loss, particularly of steel, are sufficiently great to warrant further comment. These data from exposures at Kearny, N. J. and New York City are presented graphically in Figs. 1 and 2.

TABLE II.—AVERAGE WEIGHT LOSSES AND STANDARD DEVIATIONS OF FOUR SPECIMENS OF STEEL AND OF ZINCE EXPOSED ONE YEAR AT SEVERAL LOCATIONS. (AVERAGE 1-YR WEIGHT LOSSES AND STANDARD DEVIATIONS, GRAMS, OF SAMPLES OF STEEL AND OF ZINC EXPOSED IN THE YEARS SHOWN.)

Location	Steel								Zinc							
	1948		1949		1950		1951		1948		1949		1950		1951	
	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ	$\bar{X}$	σ
State College, Pa.	6.9	0.07	6.3	0.07	8.8	0.15	6.1	0.05	0.36	0.05	0.28	0.01	0.24	0.01	0.27	0.01
Middletown, Ohio	9.1	0.12	9.2	0.09	10.9	0.15	8.2	0.05	0.32	0.03	0.30	0.01	0.28	0.02	0.27	0.01
South Bend, Pa.	10.8	0.12	10.0	0.05	13.8	0.12	9.8	0.09	0.60	0.07	0.33	0.01	0.53	0.02	0.37	0.01
Sandy Hook, N. J.	15.5	0.09	13.2	0.09	16.8	0.16	...	...	0.49	0.02	0.52	0.01	0.46	0.01	0.47	0.01
Kearny, N. J.	34.5	0.95	19.1	0.21	39.5	2.5	20.1	0.46	1.11	0.05	0.77	0.01	0.79	0.01	0.79	0.01
New York, N. Y.	...	...	19.5	0.17	35.3	0.22	17.6	0.20	...	...	1.20	0.04	1.07	0.01	1.05	0.01
New York, N. Y.[a]	41.9	0.19	50.7	0.07	39.1	0.96	44.0	1.5	1.16	0.09	1.44	0.02	1.20	0.02	1.04	0.02
Kure Beach, N. C.[b]	7.8	0.33	11.5	1.54	10.6	0.06	12.2	0.37	0.53	0.04	0.50	0.01	0.43	0.02	0.43	0.03

[a] These samples were exposed in the fall; all other samples were exposed in the spring.
[b] These samples were exposed approximately 800 ft from the ocean.

Discussion of Data:

Geographically, the test sites at Kearny and New York City are only a few miles apart. One is at ground level in the center of industrial activity in New Jersey; the other is on the roof of the 17-story, Port of New York Authority Building. In addition to the annual spring exposures at all locations, a series of specimens was exposed in New York City in the fall of each year.

With reference to the zinc sample,[2] it will be noted from Fig. 1 that those exposed at New York City were corroded more severely than those exposed at Kearny. Furthermore, those samples exposed in the fall of the year at New York City were corroded more severely than those exposed in the spring, except for the exposures made in 1951.

The samples exposed at Kearny in the spring of 1948 corroded essentially the same as those exposed in the fall of 1948 at New York City. However, samples subsequently exposed at Kearny corroded less than the samples exposed in 1948, but the attack at New York City increased in 1949 and then decreased during 1950 and 1951.

The comparison of weight losses of samples exposed at New York City in the spring and fall also is interesting. The spring exposures of 1949 and 1950 corroded less than the fall exposures, but there was no difference between the samples exposed in the spring and fall of 1951.

These differences in annual corrosion rates point out the fact that it is difficult and often misleading to attempt a statement of general conclusions regarding the corrosiveness of a particular test site. Although Kearny and New York City are geographically close together, there is a definite difference in the severity of atmospheric corrosion on specimens of zinc exposed simultaneously at the two locations. Furthermore, at New York City it has been shown that the time of the year in which the exposure is made also may have an influence on the corrosive attack. Previous work by one of the authors[3] has shown that weather conditions during the first week of exposure determined the subsequent rates of corrosion of sheet zinc exposed at a single location. Weather conditions with variations in level of atmospheric pollution could be expected to account for considerable differences in severity of corrosion.

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[2] By "sample" is meant a group of specimens.

A comparison of the weight losses of the periodically exposed samples at the several locations, as shown in Table II, indicates that at most locations the percentage variation between weight losses of steel samples from successive exposures are greater than for zinc samples exposed during the same periods. When the four, or in one case three, successive 1-yr weight losses are averaged it is seen, as shown in Table III, that the ratios of corrosiveness of the various locations are nearly the same whether determined by zinc or by steel. The greatest difference is at Kearny where the weight losses for zinc were three times those at State College, but the weight losses for steel were four times those at State College.

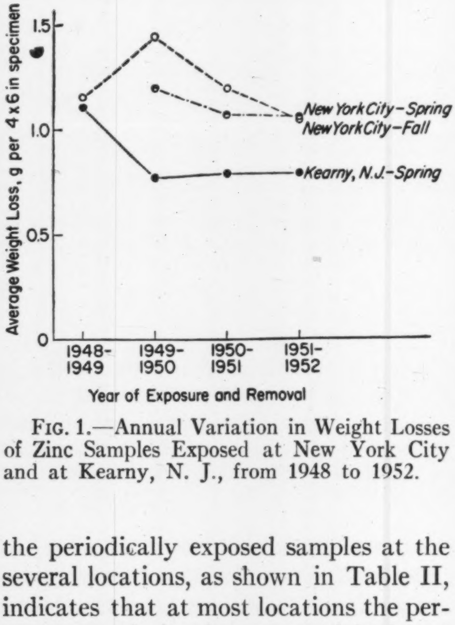

FIG. 1.—Annual Variation in Weight Losses of Zinc Samples Exposed at New York City and at Kearny, N. J., from 1948 to 1952.

The weight losses from the spring exposures of steel samples at Kearny and at New York City are in very good agreement, as shown in Table II and Fig. 2. Samples of steel exposed during succeeding springs at both locations showed periodic fluctuation of nearly 100 per cent in weight losses. This is a noteworthy example of the effect of weather immediately following exposure on the corrosion of steel. The fact that this is the case is shown by comparison of the weight losses of steel samples exposed in the fall at New York City. For example, in Fig. 2 it is seen that samples exposed in the fall of 1948 and of 1949 had weight losses over twice those of the samples exposed in the spring of 1949.

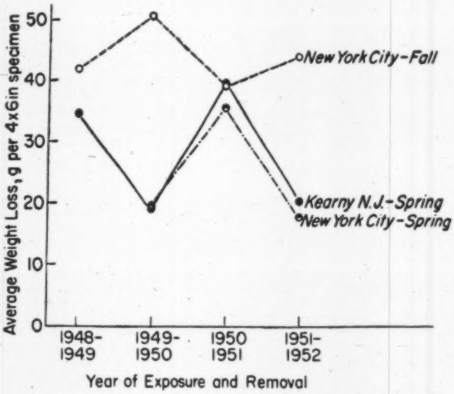

FIG. 2.—Annual Variation in Weight Losses of Steel Samples Exposed at New York City and at Kearny, N. J., from 1948 to 1952.

TABLE III.—RATIOS OF CORROSIVENESS OF ATMOSPHERES WITH STATE COLLEGE AS UNITY. (AVERAGE OF FOUR SUCCESSIVE YEARLY EXPOSURES.)

Material	State College, Pa.	Middletown, Ohio	South Bend, Pa.	Kure Beach, N. C.	Sandy Hook, N. J.	Kearny, N. J.	New York, N. Y.[a]
Zinc	1	1.3	1.5	1.5	1.7	3.0	3.8
Steel	1	1.0	1.6	1.6	2.2	4.0	3.4

[a] Spring exposures. No exposures at State College in the fall for comparison with the fall exposures at New York City.

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[3] O. B. Ellis, "Effect of Weather on the Initial Corrosion Rate of Sheet Zinc," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 152 (1949).

Similarly, specimens exposed in the fall of 1950 and of 1951 had over twice the weight losses of the specimens exposed in the spring of 1951.

Although the corrosion rate of zinc has been shown to vary with weather conditions immediately following exposure, the same factors of weather are not responsible for the variations in the corrosion rate of steel. In Fig. 1 the weight losses of zinc samples exposed at Kearny in the spring of 1949, 1950, and 1951 are very much alike; whereas, as shown in Fig. 2, the weight losses of similarly exposed steel samples varied greatly.

Summary:

Samples of zinc or of steel can be used to measure the relative corrosiveness of the atmosphere at different test sites if average weight losses of yearly exposures are used. This has been demonstrated by data from four successive 1-yr exposures at seven test sites varying from very severe to relatively mild degrees of corrosiveness.

The relative degree of severity of corrosiveness, as determined by a single exposure of zinc or of steel, could vary considerably from what is determined by the average of several successive 1-yr exposures.

REPORT OF COMMITTEE B-4

ON

ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS*

Committee B-4 on Electrical Heating, Resistance, and Related Alloys has held three meetings since the presentation of its previous report as follows: In Atlantic City, June 5 and 6, 1952; in Washington, D. C., September 25 and 26, 1952; and in New York, N. Y., January 29 and 30, 1953.

The present membership of Committee B-4 totals 68 members, of whom 40 are classified as consumers, 20 as producers, and 8 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Determining Hardness of Electrical Contact Materials (B 277 – 52 T).

Tentative Recommended Practice for:

Sublimation Testing by the Electrical Resistance Method (B 278 – 52 T).

These recommendations were accepted by the Standards Committee on December 12, 1952, and the tentative method and recommended practice will appear in the 1953 Supplement to Book of ASTM Standards, Part 2.

On April 16, 1953, the Administrative Committee on Standards accepted revisions of the Tentative Specifications for Round Chromium-Copper Wire for Electronic Devices (B 268 – 52 T). The revised specifications will appear in the 1953 Supplement to Book of ASTM Standards, Part 2.

REVISION OF TENTATIVE

The committee recommends that the Tentative Specification for High-Resistivity, Low-Temperature-Coefficient Wire (**B 267 – 52 T**)[1] be revised as follows and continued as tentative:

Section 7.—Revise to read as follows:

> The change in resistance with change in temperature shall be within the following limits, and at no point within these ranges shall the temperature coefficient exceed the corresponding value.
>
> ±0.00002 ohms per ohm per deg Cent between −65 and +125 C
>
> ±0.00003 ohms per ohm per deg Cent between +125 and +250 C

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following specifications be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

17 Per Cent Chromium-Iron Alloy for Sealing to Glass (B 256 – 51 T),[1] and

28 Per Cent Chromium-Iron Alloy for Sealing to Glass (B 257 – 51 T).[1]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 2.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating Materials (C. L. Raynor, chairman).—Additional work has been done leading to improvements in the Specification B 267. Revisions were made on Specifications for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical-Heating Elements (B 82 – 52), and Specifications for Drawn or Rolled Alloy, 60 per cent Nickel, 16 per cent Chromium, and Balance Iron, for Electrical-Heating Elements (B 83 – 52), primarily from the standpoint of resistivity *versus* size tolerances. Adequate revisions have been proposed for letter ballot. A paper on "Life Testing of Heating Alloys" was presented by Mr. Charles Guettel of the Driver Harris Co. at the January meeting.

Subcommittee V on Wrought and Cast Alloys for High Temperature Use (E. Edmunds, chairman) has reviewed the department of commerce specification N-80 and has raised question on the allowance of 80–20 alloys in carburizing, nitriding, and carbonitriding applications. This group feels that there are no provisions for 25–30 alloys under carburizing conditions and these comments have been brought to the attention of the originator of this specification.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman) is working on a revision of the Tentative Method of Test for Modulus of Elasticity of Thermostat Metals (B 223 – 51 T). In addition, the group has reviewed and proposed changes in Methods B 106, Methods of Testing Thermostat Metals.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman).—The subcommittee introduced work on clad material into the present wire section and are reviewing specification drafts on nickel-clad steel, aluminum-clad steel, nickel-plated steel, and aluminum-plated steel. In addition, this group made revisions in the specification on chromium-copper wire (B 268) and made a general survey of other round wire items from a physical point of view to establish a foundation upon which more detailed specifications could be formed.

The mica group of this subcommittee is concerned with improving the general specifications for mica stampings. This work is being done in coordination with Committee D-9 on Electrical Insulating Materials. The group is also working on the gas content and general test methods on the mica prior to the fabrication of stampings.

Section A on Nickel-Cathode Materials.—In the work on Tentative Specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (B 239 – 49 T), revisions were made of alloy grading and commercial grade 330 was added as ASTM No. 7.

Activity continues on a standard triode to overcome some of the deficiencies of the present diode. Three laboratories are working on this project.

The data group has continued work on performance analysis of newer melts Nos. 86, 87, 88, and 89, and has made recommendations on the subject of nickel stock piling in answer to inquiries from the Munitions Board. Particular investigations were made with respect to cobalt content in the nickel as related to electronic tube performance. Latest evidence, however, does not bear out the earlier apparent correlation between the cobalt content and tube performance.

Active programs continue on gas analysis and sublimation, and progress has been made in the chemical group in coordination with Committee E-3 on Chemical Analysis of Metals, where agreement has been reached on methods of test for manganese, cobalt, titanium, iron, and copper in cathode nickel. The physical test group has recommended changes in Tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 - 52 T) and Specifications B 239. Activity continues on improved methods for micrometer measurement in conjunction with Committee E-1 on Methods of Testing.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

At the Washington meeting, Mr. Ryder of Bell Telephone Laboratories presented a talk on the subject of transistors.

Subcommittee IX on Method of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman) continues the study of the behavior of high-temperature-resistant alloys in atmosphere and oxygen as related to the disassociation pressure of nickel oxide. In addition, field tests are in progress on 30-15 alloy in nitriding and carburizing atmospheres; also, fatigue tests under carburizing conditions and continued study of "green rot."

Subcommittee X on Contact Materials (F. E. Carter, chairman).—General agreement was reached on a standard type of relay for "surety of making contact." Variations in contact current and occasional insulation of contacts by films and dirt, with resulting test variations, has resulted in work on several proposed test devices. At the September meeting Mr. H. E. Stauss of the Naval Research Laboratory spoke on the effects of circuit constant atmosphere on gas pressures on contact performance. In consideration of improved contact life, the importance of the radius to contact diameter of 4 to 1 was seen significantly better than 3.1 to 1.

A new complete "Bibliography on Electrical Contacts" was issued in November, 1952, as *STP No. 56-G.* The June meeting welcomed the talk by Mr. Holm[3] on welding of contacts.

This subcommittee also improved its programming with respect to microcurrents, developed the method of test for hardness, and continued its work on thermal conductivity.

The report has been submitted to letter ballot of the committee, which consists of 68 members; 54 members returned their ballot, of whom 49 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

STANTON UMBREIT,
Secretary,

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[3] Else Holm and Ragnar Holm, "The Fundamentals of the Welding of Electric Contacts Under Heavy Current Conditions," ASTM BULLETIN, No. 188, February, 1953 p. 39 (TP 31).

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held three meetings during the year: at Philadelphia, Pa., on November 19, 1952, and at New York, N. Y., on June 25, 1952 and March 18, 1953. At these three sessions the Advisory Committee and Subcommittees W-1, W-2, W-3, W-4 also met; Subcommittees F-1, G-1, and G-3 met at the November and March sessions.

During the year, 15 members were added to the committee and there were 16 removals. At the present time the committee consists of 135 members, of whom 114 are voting members; 52 are classified as producers, 41 as consumers, and 21 as general interest members.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1952 Annual Meeting, Committee B-5 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Specification for:

Copper Rod, Bar, and Shapes (B 133 – 52 T).

Tentative Revision of Standard Specifications for:

Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 52), and

Manganese Bronze Rod, Bar, and Shapes (B 138 – 52).

These recommendations were accepted by the Committee on Standards on September 5, 1952.

On October 23, 1952 the Standards Committee accepted the following recommendation of Committee B-5:

Revision of Tentative Specification for:

Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 52 T).

The revised tentative specifications and tentative revisions appear in the 1952 Book of ASTM Standards, Part 2.

Recommendations Affecting Standards

The committee is submitting one new tentative specification, revisions in one tentative specification, and tentative revisions in 10 standards. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

Activities of Subcommittees

The following is the report of the several subcommittees during the year. The actions on standards are given in detail in the Appendix.

Subcommittee W-1 on Plate, Sheet, and Strip (S. A. Rosecrans, chairman) recommended at the June meeting that revisions be incorporated in Tentative Specification B 122.

At the November meeting, the subcommittee recommended revisions be incorporated in Tentative Specification B 248 for General Requirements. Tentative revisions were recommended in four standard specifications: B 130, B 131, B 152, and B 171.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29-July 3, 1953.

[1] See p. 205.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Among the subjects being studied by the subcommittee are the preparation of yield strength requirements for material covered by Specification B 11 for Copper Plates when used by The American Society of Mechanical Engineers; the preparation of another specification for copper-beryllium alloy; the correlation of Rockwell hardness and tensile strength values; the simplification of chemical requirements for copper-silicon alloys; temper limitations for cold-rolled copper; and the preparation of requirements for: 10 per cent nickel silver to be added to Specification B 122; manganese brass resistance welding; and tin-coated copper.

Subcommittee W-2 on Rods, Bars, and Shapes (J. D. MacQueen, chairman) recommended at the June meeting that revisions be incorporated in Tentative Specification B 133, and that Standard Specification B 138 be tentatively revised. A task group (G. H. Harnden, chairman) prepared the revisions for tensile requirements in Specification B 133. A task group (G. C. Mutch, chairman) prepared the dimensional tolerance requirements for hot-forged material in the tentative revisions for Specification B 138.

At the November meeting the subcommittee recommended tentative revisions for three standard specifications: B 21, B 138, and B 150. A task group (H. C. Ashley, chairman) prepared the requirements for mercurous nitrate test specimens in these tentative revisions.

Among the subjects being considered by the subcommittee are the preparation of a specification for copper and copper-alloy die forgings (hot-pressed); yield strength requirements for material covered by Specification B 12 for Copper Rods when used by the ASME; and provision for dimensional tolerances for hot-forged material in Specification B 138. The subcommittee is also considering the revision of tensile requirements in Specification B 133; the simplification of chemical requirements for copper-silicon alloys; the deletion of provision for machined bend test specimens in Specification B 187; and the revision of chemical requirements for rod of Type OF copper.

Subcommittee W-3 on Wire and Wire Rod (W. D. France, chairman) is studying the simplification of chemical requirements in Specification B 99; the preparation of another specification for copper-beryllium alloy; requirements for 10 per cent nickel silver wire to be added to Specification B 206; and requirements for copper flat products with finished edges.

Subcommittee W-4 on Pipe and Tube (R. S. Pratt, chairman) recommended tentative revisions in Standard Specification B 111 at the June meeting, and, at the November meeting, tentative revisions for standard specifications B 14, B 43, and B 111. A task group (H. C. Ashley, chairman) prepared the requirements for mercurous nitrate test specimens in the tentative revisions for Specifications B 43 and B 111. The subcommittee recommends a proposed new Tentative Specification for Seamless Copper Tube for Refrigeration Field Service, prepared by a task group (J. S. Rodgers, chairman).

Among the subjects under consideration in the subcommittee are the review of temper requirements in Specification B 75 for seamless copper tubes, and Rockwell hardness requirements in tube specifications. For Specification B 111, the subcommittee is studying the revision of dimensional tolerances, revision of requirements for the expanding test, the deletion of 80-20 copper-nickel alloy, and revision of requirements for copper content in aluminum bronze. Yield strength requirements are being prepared for the following specifications for tube and pipe when used by the ASME: B 13, B 42, B 43, B 75, and B 111. The subcommittee is also studying the preparation of revised chemical requirements for Type OF copper in Specification B 75; the elimination of the microscopic examination for cuprous oxide in Type DHP copper in Specifications B 42, B 68, B 75, and B 88; the preparation of requirements for new pipe-size schedules for copper, admiralty metal, copper-silicon alloys and copper-nickel alloys; and revised limits for lead in Alloy No. 3 in Specification B 135. For Specification B 251 for General Requirements, the subcommittee is considering revision of lot-size requirements for sampling large-size pipe.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman; M. L. Steinbuch, secretary) recommended that Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloys for Sand Castings B 208 - 49 T be continued as tentative.

Among the subjects under consideration in the subcommittee are the revision of chemical requirements in Specification B 198; the preparation of tables for chemical and physical requirements in Specification B 271; and, for Recommended Practice B 208, preparation of an Appendix covering a code of procedure for the inspection of copper-base alloy sand castings. The subcommittee is also preparing a specification for nickel-tin bronze castings.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman; J. P. Guerard, secretary) is preparing recommended practices for the measurement of dimensions such as thickness, width, diameter, and length. The subcommittee is reviewing Tentative Methods E 91 to determine what changes are desirable to permit the use of these methods for estimating the grain size of copper and copper alloys, and is continuing its work on the analysis of previous studies on the determination of grain size.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman), in cooperation with Subcommittee W-1, recommended for Specification B 248 the revision of width tolerances for slit metal and slit metal with rolled edges, and the addition of length tolerances for square-sheared and sawed metal. For Specification B 251, the subcommittee recommended to Subcommittee W-4 the revision of sampling requirements, the addition of a definition for average tube diameter, and the deletion of straightness tolerances for copper water tube.

Among the subjects being considered by the subcommittee are a stated temperature for making dimensional measurements; for Specification B 248, the deletion of dimensional tolerances for flat products over 1 in. thick; and for Specifications B 135 and B 188, the revision of straightness tolerances.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) recommended the detailed alphabetical subject index to contents of the special volume of ASTM Standards on Copper and Copper Alloys; that the committee sponsor the publication of an extensive ASTM monograph on the mechanical properties of phosphor bronzes based on a cooperative investigation by Bell Telephone Laboratories Inc. and The Riverside Metal Co.; and the elimination of such terms as "class" and "grade" for designating alloys in standards.

Among the subjects under consideration in the subcommittee is the preparation of a classification of wrought copper alloys.

This report has been submitted to letter ballot of the committee, which consists of 114 voting members; 100 returned their ballots, of whom 97 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specification for:

Copper and Copper-Alloy Die Forgings (Hot-Pressed) (B 283 – 53 T).

Revision of Tentative Specifications for:

General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 52 T), and
General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 52 T).

Tentative Revision of Standard Specifications for:

Copper Plates for Locomotive Fireboxes (B 11 – 49),
Copper Rods for Locomotive Staybolts (B 12 – 52),
Seamless Copper Water Tube (B 88 – 51),
Seamless Brass Tube (B 135 – 52),
Manganese Bronze Rod, Bar, and Shapes (B 138 – 52), and
Copper-Alloy Condenser Tube Plates (B 171 – 52).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentatives together with the tentative revisions of the standards appear in the 1953 Supplement to Book of ASTM Standards, Part 2.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1952 Book of ASTM Standards, Part 2.

NEW TENTATIVE

The committee recommends the following new specification for publication as tentative:

Tentative Specification for Seamless Copper Tube for Refrigeration Field Service.[1]

REVISION OF TENTATIVE

The committee recommends that the following tentative specification be revised as indicated below and continued as tentative.

Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 - 52 T):

Table IV.—Revise to read as shown in the accompanying Table I.

Section 8(d).—Add references to length tolerances for square-sheared metal and sawed metal in new Tables XIV and XV (accompanying Tables II and III) indicating that the new tables are applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.

New Tables.—Add new Tables XIV

TABLE I.—WIDTH TOLERANCES FOR SLIT METAL AND SLIT METAL WITH ROLLED EDGES.

(Applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.)

Width, in.	Width Tolerances[a] plus and minus, in.			
	For Thicknesses 0.004 to 0.032 in., incl	For Thicknesses over 0.032 to 0.125 in., incl	For Thicknesses over 0.125 to 0.188 in., incl	For Thicknesses over 0.188 to 0.375 in., incl
2 and under	0.005	0.010	0.012	0.015
Over 2 to 8, incl.	0.008	0.013	0.015	0.015
Over 8 to 20, incl.	1/64	1/64	1/64	1/32

[a] If tolerances are specified as all plus or all minus, double the values given.

TABLE II.—LENGTH TOLERANCES FOR SQUARE-SHEARED METAL IN ALL WIDTHS 120 IN. AND UNDER.

(Applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.)

Length, in.	Length Tolerances[a], plus and minus, in.		
	For Thicknesses up to 1/16 in., incl	For Thicknesses over 1/16 to 1/8 in., incl	For Thicknesses over 1/8 in.
20 and under	1/32	3/64	1/16
Over 20 to 36, incl	3/64	3/64	1/16
Over 36 to 120, incl	1/16	1/16	1/16

[a] If tolerances are specified as all plus or all minus, double the values given.

TABLE III.—LENGTH TOLERANCES FOR SAWED METAL.

(Applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.)

NOTE.—The following tolerances are all plus; if all minus tolerances are desired, use the same values; if tolerances are desired plus and minus, halve the values given.

Width, in.	Length Tolerance, in.
12 and under	1/4
Over 12 to 120, incl	1/4

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[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 2.

and XV to read as indicated in the accompanying Tables II and III.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends revisions of 10 standards for publication as tentative as indicated below:

Standard Specification for Seamless Brass Boiler Tubes (B 14 - 49):

Section 4.—Revise the requirement for copper content from the present "68.5 min" to read "68.5 to 71.5" per cent.

Standard Specification for Naval Brass Rod, Bar, and Shapes (B 21 - 52):

Section 6.—Revise to read "The test specimens, cut at least . . . ."

Standard Specification for Seamless Red Brass Pipe, Standard Sizes (B 43 - 52):

Section 7.—Revise to read "The test specimens, cut 6 in. in length . . . ."

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 52):

Section 10.— Revise to read "The test specimens, cut 6 in. in length . . . ."

Standard Specifications for:
Gilding Metal Strip (B 130 - 52), and
Gilding Metal Bullet Jacket Cups (B 131 - 52):

In the title and throughout the specifications revise "Gilding Metal" to read "Commercial Bronze."

Standard Specifications for:
Manganese Bronze Rod, Bar, and Shapes (B 138 - 52), and
Aluminum Bronze Rod, Bar, and Shapes (B 150 - 52):

Section 6.—Revise as indicated above for Section 6 in Specification B 21.

Standard Specification for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 - 52):

Table II.—Delete "B" scale Rockwell hardness values.

Standard Specification for Copper-Alloy Condenser Tube Plates (B 171 - 52):

Table I.—For copper-nickel alloy delete tin requirement of "1.5 max."

Section 7.—Delete and renumber subsequent sections accordingly.

Section 8.—In Paragraph (*a*) delete the words "except bending properties."

Delete Paragraph (*b*).

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS*

Committee B-6 held two meetings during the year: one in New York, N. Y., on June 25, 1952, and one in Detroit, Mich., on March 4, 1953, with an attendance of 41 and 36, respectively.

There is a balance of $2697.56 in the funds of Committee B-6 as of December 19, 1952.

At the present time, the committee consists of 90 members, of whom 42 are classified as producers, 24 as consumers, and 24 as general interest members.

REVISION OF STANDARD, IMMEDIATE ADOPTION[1]

The committee recommends for immediate adoption[1] the following revision in Standard Specifications for Zinc-Base Alloy Die Castings (**B 86 – 52**)[2] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Table I.—Change the maximum limit for copper for Alloy XXIII from 0.10 to 0.15 per cent.

This specification change was submitted to letter ballot a year ago at which time 79 members returned their ballots, of whom 53 voted affirmatively and 1 negatively. In view of the one negative vote, the proposed revision was removed from the 1952 report and reconsidered by both Subcommittee II and Committee B-6. Now that the revision has been reworded in an attempt to satisfy the negative vote, the recommendation has been resubmitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Advisory Committee (W. Babington, chairman) reported that the following two changes were desirable in the By-Laws of Committee B-6:

1. Article III, Section 1, Part 4, "The Secretary-Treasurer of the Society, *ex-officio*" should be changed to "A representative of the Staff of the Society, *ex-officio*," since the Society does not have a Secretary-Treasurer.
2. Article VI, Section 4, "If a member of a subcommittee or his authorized representative fails to attend a meeting of the subcommittee for two consecutive years ...." should be changed to read ".... for three consecutive meetings ...." to make it consistent with Article V, Section 3.

D. L. Colwell was appointed as the official representative of Committee B-6 on Subcommittee VII (Codification of Light Alloys) of Committee B-7.

Committee B-6 requested that the Society schedule winter meetings in January, preferably during the last two weeks, rather than the customary first week in March.

Subcommittee I on Aluminum-Base Die Casting Alloys (E. V. Blackmun, chairman) reported that the first attempt to

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] This recommendation failed of approval at the Annual Meeting; see also Editorial Note.

[2] 1952 Book of ASTM Standards, Part 2.

[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

TABLE I.—EFFECT OF EXPOSURE TO THE ATMOSPHERE ON THE MECHANICAL PROPERTIES OF 8 PER CENT MAGNESIUM ALLOY (G8) TEST BARS AFTER 5 YEARS[a] EXPOSURE.

(Tested only by Laboratory No. 1)

Specimen	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength (0.2 per cent Offset), psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength (0.2 per cent Offset), psi.	Elongation in 2 in., per cent
EXPOSED ON ROOF OF BELL TELEPHONE LABORATORIES, NEW YORK, N. Y.						
CAST IN GOOSENECK MACHINES						
No. 1	35 400	25 000	4.0	40 300	27 700	4.0
No. 2	36 400	26 000	3.5	42 200	27 700	4.5
No. 3	34 800	26 000	3.0	41 300	27 300	4.5
No. 4	37 000	25 400	3.5	41 700	28 700	4.0
No. 5	35 700	26 200	3.0	39 200	28 700	4.5
Average	35 900	25 700	3.4	40 900	28 000	4.3
CAST IN COLD CHAMBER MACHINES						
No. 1	...	...	...	44 900	29 200	5.5
No 2	41 200	25 500	5.5	36 500	28 100	4.0
No. 3	38 500	24 700	5.0	44 700	28 700	6.0
No. 4	40 100	24 500	4.5	41 800	28 900	5.0
No. 5	40 100	24 500	5.0	47 200	28 900	6.0
Average	40 000	24 800	5.0	43 000	28 800	5.3
EXPOSED AT SANDY HOOK, N. J.						
CAST IN GOOSENECK MACHINES						
No. 1	38 800	23 700	5.5	39 900	25 500	4.5
No. 2	39 400	22 900	5.5	41 900	25 800	5.0
No. 3	37 600	24 400	5.0	41 500	25 800	5.0
No. 4	39 500	22 400	5.5	43 600	25 800	5.5
No. 5	38 100	22 400	5.0	40 600	26 100	4.5
Average	38 700	23 200	5.3	41 500	25 800	4.9
CAST IN COLD CHAMBER MACHINES						
No. 1	40 300	23 500	5.5	46 000	26 300	7.5
No. 2	40 800	23 800	5.5	46 800	26 100	7.5
No. 3	39 000	22 600	5.0	47 800	26 600	7.5
No 4	38 100	22 900	5.0	39 200[b]	25 200[b]	4.0[b]
No. 5	39 400	22 600	5.0	45 500	26 900	7.5
Average	39 500	23 100	5.2	46 500	26 500	7.5

[a] One and two year data were in *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 191 (1948).

[b] Considerable porosity—not included in averages.

TABLE II.—EFFECT OF EXPOSURE TO THE ATMOSPHERE ON THE MECHANICAL PROPERTIES OF 9.5 PER CENT SILICON, 0.5 PER CENT MAGNESIUM ALLOY (SG100A) TEST BARS AFTER 5 YEARS[a] EXPOSURE.

(Tested only by Laboratory No. 1)

Specimen	Manufacturer A			Manufacturer B		
	Tensile Strength, psi.	Yield Strength (0.2 per cent Offset), psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Yield Strength (0.2 per cent Offset), psi.	Elongation in 2 in., per cent
EXPOSED ON ROOF OF BELL TELEPHONE LABORATORIES, NEW YORK, N. Y.						
COMMERCIAL PURITY GRADE						
No. 1	39 500	25 000	3.0	43 100	30 200	2.0
No. 2	40 700	24 300	2.5	44 500	30 200	2.0
No. 3	38 200	27 400	2.5	44 100	29 200	2.5
No. 4	39 500	27 400	2.5	43 000	29 900	2.0
No. 5	...	....	...	43 200	31 000	2.0
Average	39 500	26 000	2.5	43 600	30 100	2.1
HIGH PURITY GRADE						
No. 1	40 300	24 700	3.5	41 800	25 700	2.5
No. 2	40 800	24 900	3.5	43 900	25 500	2.5
No. 3	...	24 500	3.0	42 100	25 100	2.0
No 4	41 700	24 500	3.5	43 000	25 900	2.0
No. 5	40 300	21 900	3.5	41 800	25 100	2.0
Average	40 800	24 100	3.4	42 500	25 500	2.2
EXPOSED AT SANDY HOOK, N. J.						
COMMERCIAL PURITY GRADE						
No. 1	38 300	25 600	2.5	42 900	28 300	2.0
No. 2	40 300	23 900	2.5	42 200	29 400	2.0
No. 3	41 400	27 000	2.5	40 700	29 400	1.5
No. 4	42 500	26 000	2.5	43 900	29 600	2.0
No. 5	40 600	25 600	2.5	42 300	27 900	2.0
Average	40 600	25 600	2.5	42 400	28 900	1.9
HIGH PURITY GRADE						
No. 1	43 000	23 300	5.0	40 100	24 600	2.5
No. 2	41 900	25 400	5.0	41 500	26 100	2.5
No. 3	41 500	25 200	4.5	41 100	26 100	2.5
No. 4	42 700	24 900	4.5	41 800	24 900	2.5
No. 5	37 000	24 500	4.5	41 500	26 300	2.5
Average	41 200	24 700	4.7	41 200	25 600	2.5

[a] One and two year data were in *Proceedings*, Am Soc. Testing Mats., Vol. 48, p. 191 (1948).

prepare a tabulation to guide users in the selection of the proper die-casting alloy for a particular application, and at making revisions in the typical mechanical properties table in the Appendix of Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 52 T), had produced some negative votes, and so both tables were referred back to the Task Group for further study.

About four years ago, the zinc limit in alloy SC84A was raised from 0.6 to 1.0 per cent on the basis of data that were accumulated specifically for that

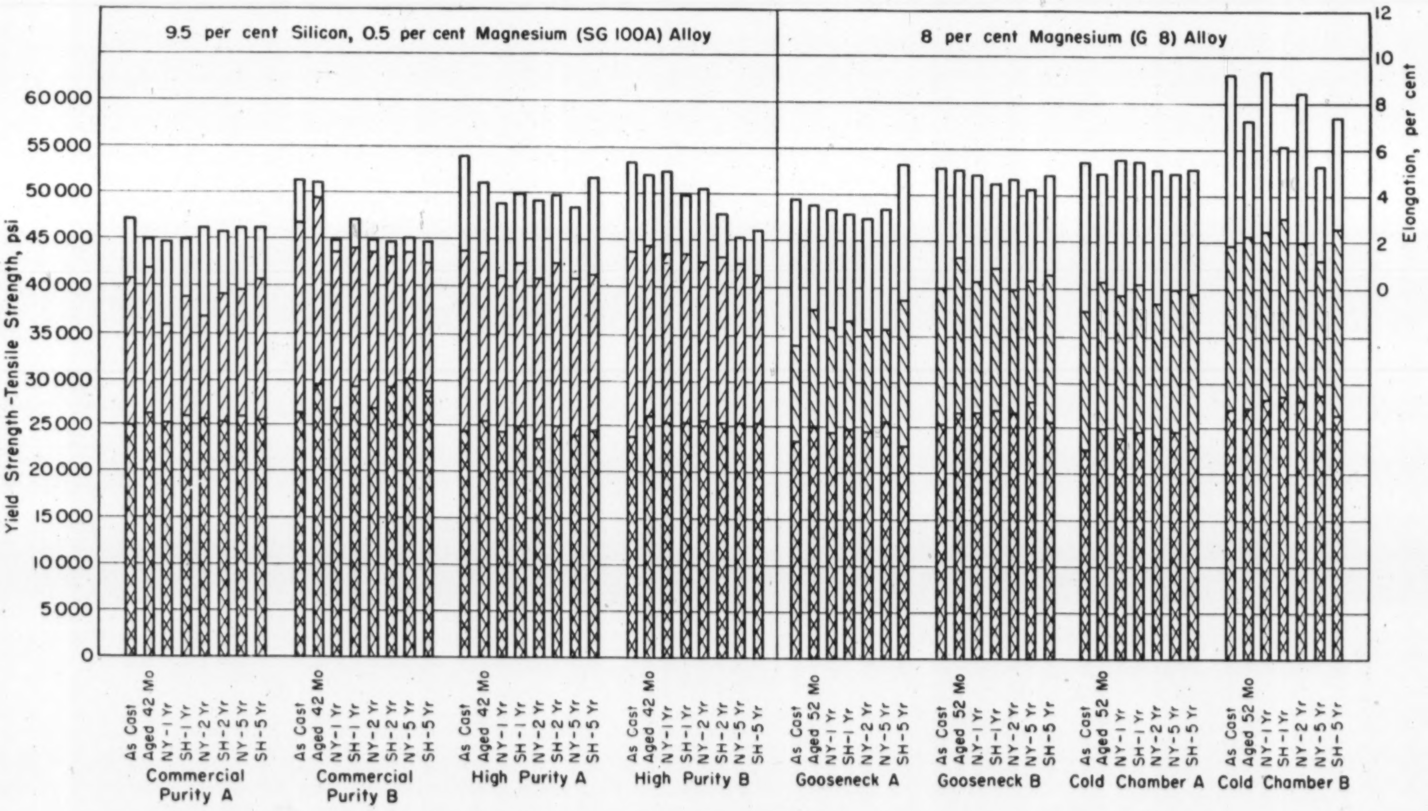

FIG. 1.—Effect of 1-, 3- and 5-yr Exposures on the Mechanical Properties of G8 and SG100A Alloys.

purpose. Unfortunately, these results were not published, but the oversight is now being corrected with the incorporation of a report on "The Effect of Zinc Content on the Mechanical Properties of the SC84A Aluminum Die-Casting Alloy" as an Appendix to this report.[4]

Subcommittee II on Zinc-Base Die Casting Alloys (A. E. Weiss, chairman) recommended that the permissible copper limit in Alloy XXIII of Standard Specifications for Zinc-Base Alloy Die Castings (B 86 – 52) be increased from 0.10 to 0.15 per cent.[5]

Subcommittee V on Exposure and Corrosion Tests (Sam Tour, chairman) reported that the tests on samples of aluminum alloys G8 and SG100A after five years of exposure at Sandy Hook, N. J., and at New York City have been completed. The latest results are reported in Tables I and II, and the combined results of the 1-, 2-, and 5-yr exposures are reported in bar chart form in Fig. 1. The lowest and median points of the bar charts indicate yield and tensile strengths, respectively. The upper point refers to the per cent elongation as indicated by the scale on the right-hand side of the figure.

The subcommittee reports also the initiation of a new series of exposure tests on aluminum alloy SC84A with various amounts of zinc from low to as high as 2 per cent. Test bars are being exposed at the 80- and 800-ft test sites at Kure Beach, N. C., New York City, and Columbus, Ohio, test sites for 1-, 3-, 6-, and 12-yr periods.

Subcommittee VIII on Brass Die Casting Alloys (J. C. Fox, chairman) reported an error in the yield strength of alloy ZS144A in Standard Specifications for Copper-Base (Brass) Alloy Die Castings (B 176 – 52). A typical yield strength of 30,000 psi is shown, whereas this should be 50,000 psi.

A Task Group has been appointed to study the effect of iron content in alloy ZS331A in the range from 0.10 to 0.50 per cent with the aim of raising the iron limit in the specification for this alloy from 0.15 to 0.25 per cent if it can be done without producing any deleterious effects.

Subcommittee IX on Die Casting Processes (A. E. Martin, chairman) reported that a paper entitled "An Analytical Study of the Die Casting Process" by Bruno Sachs was recommended to the Society for publication in the BULLETIN.[6] It was the feeling of the committee that the paper represented a forward step in the fund of basic knowledge on the die casting process and that this paper, in conjunction with those by Jacobi[7] and by Babington and Kleppinger,[8] would serve to stimulate additional discussion and eventually lead to further advances in the process.

This report has been submitted to letter ballot of the committee, which consists of 90 members; 84 members returned their ballots, of whom 76 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

G. L. WERLEY,
Secretary.

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[4] See p. 212.

[5] See p. 211.

[6] ASTM BULLETIN, No. 192, September, 1953, p. 27 (TP117).

[7] Edward Jacobi, "Flow Calculations for Die Casting Applied to the ASTM Committee B-6 Test Casting Die," ASTM BULLETIN, No. 166, May, 1950, p. 65 (TP127).

[8] W. Babington and D. H. Kleppinger, "Aluminum Die Castings—The Effect of Process Variables on Their Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 169 (1951).

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-6 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specifications for Zinc-Base Alloy Die Castings (B 86 – 52) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on September 9, 1953, and the tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 2.

ADDENDUM TO B-6 REPORT

The data supporting the proposed increase in permissible maximum copper content of Alloy XXIII of Standard Specifications for Zinc-Base Alloy Die Castings (B 86–52) from 0.10 to 0.15 per cent has appeared in the minutes of Subcommittee II of B-6 and in the minutes of Committee B-6.

Attention has been called, by the one negative voter, to the fact that the supporting data for this change has not been included in the annual report. Committee B-6 wishes to correct this oversight herewith and to present a summary of these supporting data, furnished by Mr. G. H. Werley of the New Jersey Zinc Co. of Pennsylvania for inclusion with the 1953 Annual Report.

EFFECT OF INCREASE IN COPPER CONTENT OF ALLOY XXIII (B-86)

	Alloy XXIII Plus Copper Additions			
	0 per cent Copper	0.1 per cent Copper	0.3 per cent Copper	0.5 per cent Copper
As Cast				
Tensile strength, psi	42 800	43 400	44 200	44 500
Impact strength, ft-lb, ¼-in. bars	25	26	25	26
Brinell hardness	82	82	83	85
Per cent elongation in 2-in.	4	5	3	3
After Exposure to Steam at 95 C for 10 Days				
Tensile strength, psi	33 800	34 900	35 500	36 600
Impact strength, ft-lb, ¼-in. bars	29	25	21	19
Per cent elongation in 2-in.	11	9	9	10
Expansion, in. per in.	0.0007	0.0005	0.0004	0.0003
After 10 yr Indoor Aging				
Tensile strength, psi	34 700	35 800	36 600	37 000
Impact strength, ft-lb, ¼-in. bars	36	32	29	30
Brinell hardness	62	62	66	66
Per cent elongation in 2-in.	12	11	9	8
Expansion, in. per in.	0.0001	0.0001	0.0001	0.0001

APPENDIX

THE EFFECT OF ZINC CONTENT ON THE MECHANICAL PROPERTIES OF THE SC84A ALUMINUM DIE-CASTING ALLOY

At the June, 1949, meeting, a proposal was made to increase the zinc content of the SC84A alloy of ASTM Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85)[1] from 0.6 to 1.0 per cent. The history of this alloy, commonly known by its Army Specification as AXS-679, shows that until September of 1942 the zinc content was 1.0 per cent but, in a compromise action by the WPB when the silicon was increased, it was lowered to 0.6 per cent.

At that time, the control of zinc to 0.6 per cent presented no problem to the secondary alloy producers. However, the increased use of aluminum alloys with high zinc content (75S) and their return to the scrap market changed this picture, so that in 1949 the secondary alloy producers were having difficulty in meeting the 0.6 per cent zinc limit without increasing the cost of the alloy.

Discussion of the proposal to raise the zinc limit brought out two pertinent facts: (1) data were not available which showed that an increase to the proposed 1.0 per cent zinc limit would not be harmful to the mechanical properties of the SC84A alloy, and (2) a change in the requirements for SC84A would not be a complete solution to the problem unless Specification AXS-679 could be modified in a like manner.

The discussion led to the establishment of a task group charged with performing tests to determine the effect of increased zinc content on the mechanical properties of the alloy. The findings of this task group were to be used to decide the advisability of raising the zinc limit in the SC84A alloy with the hope that this move by the ASTM would be followed by revision of AXS-679 by the Ordnance Corps. This report describes the work and findings of the task group.

It was decided that SC84A alloy having five levels of zinc content should be studied and that, in order to rule out any effects due to variations in other alloying elements or impurities, all metal used in the test should come from a common heat.

The five zinc contents chosen for study were 0.25, 0.50, 1.0, 1.5, and 2.0 per cent. The 0.25 per cent zinc content was included in the work specifically to study the effects of so-called "high purity," whereas the 2.0 per cent content was included to have a composition well outside the range in question in order to establish any trend that might develop.

By referring to Fig. 1, the test procedure can be visualized. A special heat of 4500-lb SC84A alloy was melted by the Apex Smelting Co. This heat had the following nominal composition: Zn 0.20, Cu 3.50, Si 8.20, Fe 0.45, Mn 0.40, Mg 0.02, Ni 0.20, and Sn 0.01 per cent.

Nine hundred pounds of this composition were tapped off to the three casters, and zinc was added to the remainder to bring the zinc content up to 0.50 per cent where 900 lb were again tapped off. The other three zinc content alloys were prepared in a like manner by addition of zinc to the remaining master heat.

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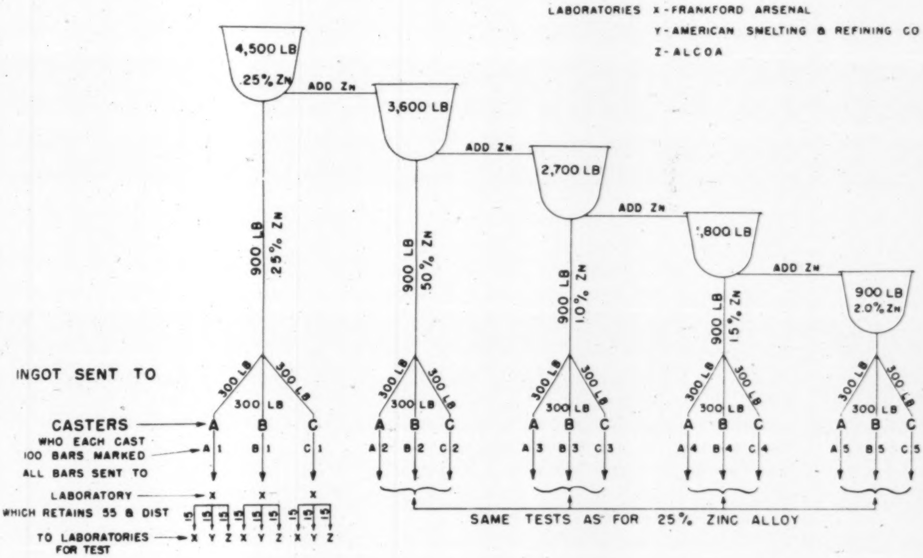
[1] 1949 Book of ASTM Standards, Part 2; Revision appears in 1952 Book of ASTM Standards, Part 2.

Each of the five lots of 900 lb of ingot was divided into three lots of 300 lb each and sent to the three die casters, Bell Telephone Laboratories, Precision Casting Co., and Stewart Die Casting Division of Stewart Warner Corp.

Each die caster made 100 test bars from each of the lots of ingot, exercising caution to prevent contamination of any of the lots and to hold casting conditions as constant as possible throughout all of the casting runs.


LABORATORIES X-FRANKFORD ARSENAL
Y-AMERICAN SMELTING & REFINING CO
Z-ALCOA
4,500 LB .25% ZN
ADD ZN
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ADD ZN
2,700 LB
ADD ZN
1,800 LB
ADD ZN
900 LB 2.0% ZN
900 LB .25% ZN
900 LB .50% ZN
900 LB 1.0% ZN
900 LB 1.5% ZN
INGOT SENT TO
300 LB
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WHO EACH CAST
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A1 B1 C1
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SAME TESTS AS FOR .25% ZINC ALLOY

FIG. 1.—Flow Chart for Investigation of Effect of Zinc on SC84A Alloy.

The casting conditions used were as follows:

	Producer A	Producer B	Producer C
Calculated pressure	6000 psi	16 600 psi	4500 psi
Plunger speed	180–200 ft per min	300 ft per min	200 ft per min
Metal temperature (pot)	1175 F	1220 F	1200 F
Die temperature	400 F	440 F	275 to 300 F

All of the bars were stamped with a code to identify producer and zinc content and were forwarded to Frankford Arsenal for distribution. In all 1500 bars were received—100 bars × 3 casters × 5 zinc levels. In the coding system the letters indicated producers and the numbers 1 to 5 indicated the respective zinc contents.

Of each 100 bars received for tests, 55 were retained for possible future tests and 45 were distributed, 15 each to the American Smelting and Refining Co., Aluminum Company of America, and Frankford Arsenal for mechanical tests and chemical analysis.

Chemical analysis was made for each lot from a composite sample drilled from the broken ends of four test bars from each lot of 15. Mechanical tests were made in accordance with ASTM standards and included determination of 0.2 per cent offset yield strength, tensile strength, and per cent of elongation.

TABLE I.—SUMMARY OF MECHANICAL TEST DATA OF SC84A ALLOY TEST BARS.

		YIELD STRENGTH psi				TENSILE STRENGTH psi				% ELONGATION			
		Lab X	Lab Y	Lab Z	Average	Lab X	Lab Y	Lab Z	Average	Lab X	Lab Y	Lab Z	Average
Producer A	A-1	19500	19800	19000	19400	41100	40600	40100	40600	3.4	3.1	3.3	3.3
	A-2	18700	19200	18400	18800	39200	39800	40900	40000	3.3	3.4	3.5	3.4
	A-3	18800	18400	18000	18400	39400	39800	39400	39500	3.0	3.3	3.0	3.1
	A-4	18900	20900	18100	19300	42100	43200	40200	41800	4.5	4.0	3.1	3.0
	A-5	19500	20100	19200	19600	41100	43900	42500	42500	3.8	4.0	3.6	3.8
Producer B	B-1	19900	21100	19400	20100	43100	42200	44600	43300	3.3	3.6	4.4	3.8
	B-2	18800	20500	19100	19500	43400	42500	44500	43500	3.8	3.8	4.5	4.0
	B-3	19400	20800	19200	19800	43900	43000	44100	43700	4.1	4.0	4.5	4.2
	B-4	19600	21000	19000	19900	42900	42400	43200	42800	3.7	3.3	4.2	3.7
	B-5	19000	20600	19200	19600	43100	42900	43700	43200	4.0	3.7	4.5	4.1
Producer C	C-1	21000	21800	22600	21800	40400	40400	42900	41200	3.1	3.2	3.4	3.2
	C-2	21900	22100	21700	21900	42200	40200	41000	41100	3.4	2.9	3.0	3.1
	C-3	21200	22900	21900	22000	42300	41600	43800	42600	4.1	3.0	4.2	3.8
	C-4	20900	22100	20900	21300	41400	40200	43200	41600	3.9	3.8	4.1	3.9
	C-5	20700	22000	21200	21300	42500	41000	42700	42000	4.2	3.9	3.9	4.0

	YIELD STRENGTH psi		TENSILE STRENGTH psi		% ELONGATION	
Grand Average	.25% Zn	20400	.25% Zn	41700	.25% Zn	3.4
	.50% Zn	20100	.50% Zn	41500	.50% Zn	3.5
	1.0 % Zn	20100	1.0 % Zn	41900	1.0 % Zn	3.7
	1.5 % Zn	20200	1.5 % Zn	42100	1.5 % Zn	3.8
	2.0 % Zn	20200	2.0 % Zn	42600	2.0 % Zn	4.0

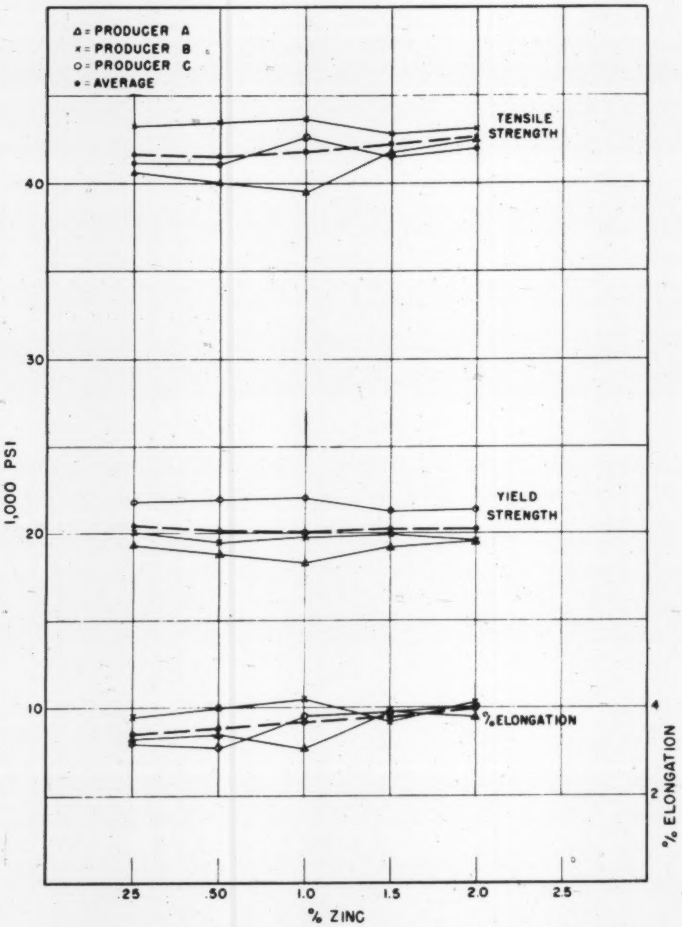

FIG. 2.—Effect of Zinc on Mechanical Properties of SC84A Alloy.

The results of the chemical tests showed that there had been no contamination of any kind and that the compositions of the lots were extremely uniform. The zinc levels actually used as determined by these analyses were 0.20, 0.55, 1.03, 1.52, and 1.95 per cent. Other elements found were in the amounts previously reported.

The results of the mechanical tests are shown in tabulated form in Table I and in graphical form in Fig. 2. In Table I, each of the values in columns headed "Lab X," "Lab Y," and "Lab Z" is the average of the 15 tests performed at that laboratory. In several instances, however, test bars with visible defects at fracture were not included in the average. The values in columns headed "Average" are the averages of the three laboratories for each producer; "Grand Average" is the average value for all casters and all laboratories.

In Table I the average values for each producer as well as the grand average for all producers have been plotted for each of the five zinc levels.

Examination of these data shows that any change in zinc content from 0.20 to 2.0 per cent in the SC84A alloy has no effect on the mechanical properties with the possible exception of a slight rise in ductility.

On the basis of data presented, an increase in the zinc content of the SC84A die casting alloy from 0.6 to 1.0 per cent will not be detrimental to the mechanical properties of the alloy.

Respectfully submitted by Task Group:

D. H. Kleppinger, *chairman.*
W. Babington,
D. L. Colwell,
M. H. Jameson,
* B. E. Sandell,
A. E. Martin, and
R. A. Quadt.

Note.—Since this report was presented to Subcommittee I, the recommendation to raise the zinc limit from 0.6 to 1.0 per cent in the SC84A composition has been carried out, and the Ordnance Corps has superseded the Specification AXS-679 by Composition 10 of Federal Specification QQ-A-591a in which the zinc limit is set at 1.0 per cent.

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* Deceased.

REPORT OF COMMITTEE B-7
ON
LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys held two meetings during the year: one at New York, N. Y., in June, 1952, and one in Detroit, Mich., in March, 1953.

The committee consists of 91 members, of whom 88 are voting members; 44 are classified as producers, 30 as consumers, and 17 as general interest members.

Fred Keller was appointed chairman of Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys and L. H. Adam was appointed Committee B-7 representative on the Advisory Committee on Corrosion, replacing J. D. Edwards, who resigned these assignments because of ill health.

REVISION OF TENTATIVES

The committee recommends that the following 14 tentatives be revised as set forth in the Appendix of this report:[1]

Tentative Specifications for:

- Magnesium-Base Alloy Sand Castings (B 80 - 51 T),
- Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 49 T),
- Aluminum and Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 - 52 T),
- Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 52 T),
- Aluminum-Alloy Drawn Seamless Tubes (B 210 - 52 T),
- Aluminum and Aluminum-Alloy Bars, Rods, and Wire (B 211 - 52 T),
- Magnesium-Base Alloy Extruded Round Tubes (B 217 - 49 T),
- Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 52 T),
- Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 50 T),
- Aluminum-Alloy Extruded Tubes (B 235 - 52 T),
- Aluminum-Alloy Pipe (B 241 - 50 T),
- Aluminum and Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Applications (B 273 - 52 T), and
- Aluminum and Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 - 52 T).

Tentative Recommended Practice for:

- Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 52 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following three tentative specifications, which have stood for two years or more without revision, be continued as tentative because it is anticipated that new alloys will be added to the specifications:

Tentative Specifications for:

- Magnesium-Base Alloy Sheet (B 90 - 51 T),
- Magnesium-Base Alloy Forgings (B 91 - 49 T), and
- Magnesium-Base Alloy Permanent Mold Castings (B 199 - 51 T).

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (**B 179 - 51 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] See p. 219.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (R. A. Quadt, chairman) was inactive during the past year, but recommends that Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 51 T) be adopted as standard without revision.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) is considering revision of Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 52 T) and Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 52 T) to add minimum yield strength requirements for use when required for such applications as pressure vessels.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman) prepared revisions of Tentative Specifications B 178 – 52 T, B 209 – 52 T, B 210 – 52 T, B 211 – 52 T, B 221 – 52 T, B 234 – 50 T, B 235 – 52 T, B 241 – 50 T, B 273 – 52 T, and B 274 – 52 T, as set forth in the Appendix to this report, and is preparing a revision of Tentative Specifications for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 52 T). Also under preparation are specifications for other forms of bus conductors and specifications for Aluminum-Alloy Standard Structural Shapes.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) prepared revisions of Tentative Specifications B 80 – 51 T, B 107 – 49 T, and B 217 – 49 T, as set forth in the Appendix to this report, and is cooperating with the American Foundrymen's Society in preparing a tabulation of the properties and characteristics of magnesium casting alloys for a future revision of Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 51 T).

Subcommittee V on Testing Light Metals (R. L. Templin, chairman) is continuing to investigate statistical sampling methods as applied to testing for mechanical properties, but is experiencing difficulty in establishing lot sizes to be used in defining a statistically satisfactory sampling method for the frequency of mechanical testing requirements of the specifications under the jurisdiction of Committee B-7.

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys (Fred Keller, chairman) changed the name and scope of the subcommittee to include anodic oxidation of magnesium alloys. Progress is being made on developing specifications for anodic coatings for various applications and of engineering data on the characteristics affecting the performance of anodic coatings on the various aluminum alloys and products.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (R. B. Smith, chairman) prepared the revision of Tentative Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 52 T), set forth in the Appendix to this report. B 275 is now being used to establish the alloy designations in all Committee B-6 specifications for die-cast metals and alloys, some of which are not light metals and alloys. Further work on codification of tempers is in progress.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of 27 aluminum and 8 magnesium alloys installed at three of the five Society atmospheric exposure test sites: for one year on top of the Port of Authority Building, New York City, for industrial atmospheric conditions; for one year at State College, Pa., for rural; and for 6 months at Kure Beach, N. C., for Atlantic Coast Marine. Arrangements have been made for erection of specimens at Point Reyes, California, for Pacific Coast Marine atmospheric conditions, but the Gulf Coast Marine site at Freeport, Tex., requires racks before specimens can be placed on test. A paper on Atmospheric Exposure of Light Metals and Alloys is appended to this report.[3]

This report has been submitted to a letter ballot of the committee, which consists of 90 members; 66 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

R. B. SMITH,
Secretary.

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[3] See p. 227.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering light metals and alloys which are referred to earlier in this report. The standards appear in their present form in the 1952 Book of ASTM Standards, Part 2.

TABLE I.—CHEMICAL REQUIREMENTS.

Alloy	Magnesium, per cent	Aluminum, per cent	Manganese, min., per cent	Zinc, per cent	Rare Earths, per cent	Zirconium, per cent	Silicon, max., per cent	Copper, max., per cent	Nickel, max., per cent	Other Impurities max., per cent
EK30A....	remainder	....	....	0.3 max.	2.5 to 4.0	0.2 min.	....	0.10	0.01	0.3
EK41A......	remainder	....	....	0.3 max.	3.0 to 5.0	0.40 to 1.0	....	0.10	0.01	0.3
EZ33A......	remainder	....	....	2.0 to 4.0	2.5 to 4.0	0.50 to 1.0	....	0.10	0.01	0.3
ZK51A......	remainder	....	....	3.6 to 5.5	....	0.50 to 1.0	....	0.10	0.01	0.3
ZK61A......	remainder	....	....	5.5 to 6.5	....	0.60 to 1.0	....	0.10	0.01	0.3

TABLE II.—TENSILE REQUIREMENTS.

Alloy	Condition	Tensile Strength, min., psi.	Yield Strength[b] (0.2 per cent offset), min., psi.	Elongation in 2 in., min., per cent
EK30A............	-T6 (solution heat treated and artificially aged)	18 000	14 000	1
EK41A............	-T5 (artificially aged only)	20 000	14 000	[a]
	-T6 (solution heat treated and artificially aged)	22 000	16 000	1
EZ33A............	-T5 (artificially aged only)	18 000	14 000	1
ZK51A............	-T5 (artificially aged only)	34 000	20 000	5
ZK61A............	-T6 (solution heat treated and artificially aged)	40 000	25 000	5

REVISIONS OF TENTATIVE SPECIFICATIONS

The committee recommends that the following 13 tentative specifications be revised as indicated below and continued as tentative.

Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 51 T):

Table I.—Add requirements for alloys EK30A, EK41A, EZ33A, ZK51A, and ZK61A as shown in the accompanying Table I.

Table II.—Delete the minimum elongation requirement of 4 per cent for alloy AZ91C-F and add requirements for alloys EK30A, EK41A, EZ33A, ZK51A, and ZK61A as shown in the accompanying Table II.

Table III.—Add alloys EK30A, EK41A, EZ33A, ZK51A, and ZK61A as shown in the accompanying Table III.

Explanatory Notes. Note 1.—Add the following:

Alloy EK30A.—This alloy has a specific gravity of about 1.79. It is used in the heat treated and aged condition. It is recommended for use at elevated temperatures, especially in the range of 300 to 500 F. This alloy will produce sound castings for pressure tightness.

Alloy EK41A.—This alloy has a specific gravity of about 1.81. It is used in the heat treated and aged and artificially aged conditions. It is recommended for use at elevated temperatures, especially in the range of 300 to 500 F. This alloy will produce sound castings for pressure tightness.

Alloy EZ33A.—This alloy has a specific gravity of about 1.84. It is used in the artificially aged condition. It is recommended for use at elevated temperatures, especially in the range of 300 to 500 F. This alloy will produce sound castings for pressure tightness.

Alloy ZK51A.—This alloy has a specific gravity of about 1.83. It is used in the artificially aged condition. This alloy produces castings combining a high yield strength with good elongation.

Alloy ZK61A.—This alloy has a specific gravity of about 1.83. It is used in the solution heat treated and artificially aged condition. This alloy produces castings of highest strength-to-weight ratio and good elongation.

TABLE III.

Alloy	Condition	Typical or Minimum	Yield Strength (0.2 per cent offset), min., psi.	Unit Deformation, in. per. in. of gage length
EK30A	-T6 (solution heat treated and artificially aged)	minimum	14 000	0.0042
EK41A	-T5 (artificially aged only)	minimum	14 000	0.0042
	-T6 (solution heat treated and artificially aged)	minimum	16 000	0.0045
EZ33A	-T5 (artificially aged only)	minimum	14 000	0.0042
ZK51A	-T5 (artificially aged only)	minimum	20 000	0.0051
ZK61A	-T6 (solution heat treated and artificially aged)	minimum	25 000	0.0058
	-T6 (solution heat treated and artificially aged)	typical	28 000	0.0063

Specifications for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 49 T):

Section 1.—Revise to read: "These specifications cover commercial magnesium-base alloy extruded bars, rods, and shapes of the compositions given in Table I.[3]"

Table I.—Delete note *a* and add requirements for alloy ZK60A as follows:

Magnesium, per cent	remainder
Zinc, per cent	4.8 to 6.2
Zirconium, min, per cent	0.45
Other Impurities, max, per cent	0.3

Table II.—Revise to read as shown in the accompanying Table IV.

Table III.—Revise title to read: "Permissible Variations in Cross-Sectional Dimensions of Alloys AZ31B, AZ61A, AZ80A, and M1A."

New Table.—Add a new Table IV titled "Permissible Variations in Cross-Sectional Dimensions of Alloy ZK60A" to read as shown in the accompanying Table V, renumbering subsequent tables accordingly.

Section 8.—Revise to read as follows:

8. Sampling.—From material having a nominal weight of less than 1 lb per lineal ft, one tension-test sample shall be selected by the Inspector from each lot weighing 500 lb or less; from lots weighing more than 500 lb, one additional sample shall be taken for each 1,000 lb or fraction thereof in excess of the first 500 lb. For material having a nominal weight of 1 lb or more per lineal ft, one tension-test sample shall be taken from each lot consisting of 500 ft, or less; from lots consisting of more than 500 ft, one additional sample shall be taken from each 1,000 ft or fraction thereof in excess of the first 500 ft.

Section 11. (*a*)—Revise to read: "Variations from the specified cross-sectional dimensions shall not exceed the amounts prescribed in Tables III and IV".

Section 11. (*b*)—Revise to read: "Bars and shapes shall not deviate from straight by an amount greater than permitted in Tables V and VI".

Table IV.—Renumber as Table V and move note *b* from last line in third column to after the word "length" in heading of third column. Revise note *b* to read: "Measured over 5 ft of length."

Explanatory Notes. Note 1.—Delete

alloy AZ80A and the sentence pertaining to it, and add the following:

> *Alloy ZK60A.*—This alloy combines high tensile strength with good ductility and has the highest compressive yield strength of any of the extrusion alloys. Minimum compressive yield strength values that can be met are given in Table VIII.

Explanatory Notes. Note 2.—In last sentence change "Table VII" to "Table IX."

TABLE IV.—TENSILE REQUIREMENTS.

Alloy and Temper	Form	Diameter or Thickness, in.	Cross-Sectional Area, sq. in.	Tensile Strength, min., psi.	Yield Strength (0.2 per cent offset), min., psi.	Elongation in 2 in., min., per cent
AZ31B-F	Bars, Rods, and Shapes	0.249 and under	All	33 000	20 000	7
		0.250 to 2.499	All	32 000	20 000	7
	Hollow Shapes	All	All	32 000	16 000	8
AZ61A-F	Bars, Rods, and Shapes	0.249 and under	All	38 000	20 000	8
		0.250 to 2.499	All	39 000	24 000	9
	Hollow Shapes	All	All	36 000	16 000	7
AZ80A-F	Bars, Rods, and Shapes	0.249 and under	All	43 000	28 000	9
		0.250 to 1.499	All	43 000	28 000	8
		1.500 to 2.499	All	43 000	28 000	6
AZ80A-T51	Bars, Rods, and Shapes	0.249 and under	All	47 000	30 000	4
		0.250 to 1.499	All	47 000	32 000	3
		1.500 to 2.499	All	47 000	32 000	2
M1A-F	Bars, Rods, and Shapes	0.249 and under	All	30 000	[a]	2
		0.250 to 1.499	All	32 000	[a]	3
		1.500 to 2.499	All	32 000	[a]	2
	Hollow Shapes	All	All	28 000	[a]	2
ZK60A-F	Bars, Rods, and Shapes	All	up through	43 000	31 000	5
	Hollow Shapes	All	5 All	40 000	28 000	5
ZK60A-T5	Bars, Rods, and Shapes	All	up through	45 000	36 000	4
	Hollow Shapes	All	5 All	46 000	38 000	4

[a] Not Required.

New Table.—Add a new Table VIII in Explanatory Notes to read as shown in the accompanying Table VI.

Table VII.—Renumber as Table IX and add alloy ZK60A as shown in the accompanying Table VII.

Specifications for Aluminum and Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 – 52 T):

Table I.—Revise to read as shown in the accompanying Table VIII. Revise the iron plus silicon limit of "0.4" in note *d* to read "0.40" per cent, and of "0.60" in note *f* to read "0.7" per cent.

Table II.—Add requirements for alloy GR40A as shown in accompanying Table IX.

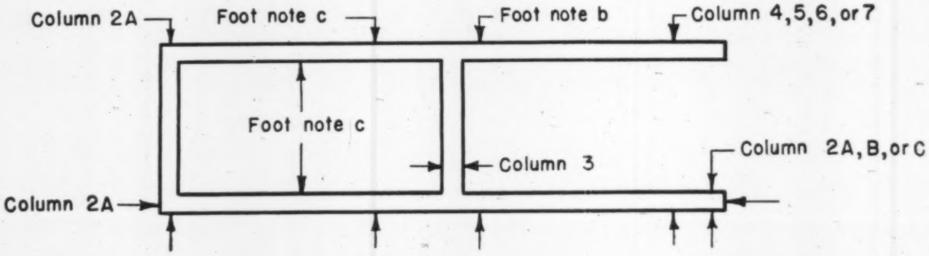
Table IV.—Revise title to read: "Permissible Variations in Thickness of Alloys GR20A, GR40A, GS11A, Clad GS11A, MG11A, and Clad MG11A Flat Sheet, Coiled Sheet, and Plate, Plus or Minus, in."

Specifications for Aluminum-Alloy Sheet and Plate (B 209 – 52 T):

Table I.—For the cladding of alloy Clad ZG62A revise the zinc range of "0.75 to 1.25" to read "0.8 to 1.3" per cent. For all alloys revise the limits in the table and notes to express values of 0.50 and less to two digits to the right of the decimal point, and values of greater than 0.50 to one digit to the right of the decimal point.

Table II.—For alloy M1A-H14 revise the tensile strength requirement of

TABLE V.—PERMISSIBLE VARIATIONS IN CROSS-SECTIONAL DIMENSIONS OF ALLOY ZK60A.



Ordered Dimension, in.[g]	Permissible Variations, plus or minus, in.[a]											
	Metal Dimensions				Space Dimensions							
	Permissible Variations from Specified Dimensions Where All of the Dimension Is Metal				Permissible Variations from Specified Dimension Where Any Part of the Dimension Is Space[b, c, f]							
	All Excepting Those Covered by Column 3			Wall Thickness[d] Completely Enclosing Space 0.11 sq in. and Over (Eccentricity)	At Dimensioned Points ¼ to ⅝ in., excl., from Base of Leg		At Dimensioned Points ⅝ to 1¼ in., excl., from Base of Leg		At Dimensioned Points 1¼ to 2½ in., excl., from Base of Leg		At Dimensioned Points 2½ in. or more from Base of Leg	
Column 1	Column 2			Column 3	Column 4		Column 5		Column 6		Column 7	
	A W/T[h] up Thru 20.0	B W/T[h] is 20.1 Thru 40.0	C W/T[h] is 40.1 and up		A[f]	B[f]	A[f]	B[f]	A[f]	B[f]	A[f]	B[f]
Up through 0.124	0.006	0.010	0.016		0.010	0.014	0.012	e	0.014	e	0.016	e
0.125– 0.249	0.007	0.010	0.016		0.012	0.015	0.014	0.023	0.016	e	0.020	e
0.250– 0.499	0.008	0.010	0.016		0.014	0.016	0.016	0.024	0.018	0.040	0.022	e
0.500– 0.749	0.009	0.010	0.016		0.016	0.017	0.018	0.025	0.020	0.041	0.026	e
0.750– 0.999	0.010	0.010	0.016		0.018	0.018	0.020	0.026	0.022	0.042	0.030	e
1.000– 1.499	0.012	0.012	0.016	10 per cent max. 0.060 min. 0.010	0.020	0.020	0.022	0.028	0.026	0.044	0.034	0.066
1.500– 1.999	0.016	0.016	0.016		0.024	0.024	0.028	0.032	0.034	0.048	0.050	0.070
2.000– 3.999	0.024	0.024	0.024		0.032	0.032	0.036	0.040	0.048	0.056	0.064	0.076
4.000– 5.999	0.034	0.034	0.034		0.042	0.042	0.050	0.050	0.064	0.064	0.088	0.088
6.000– 7.999	0.044	0.044	0.044		0.054	0.054	0.062	0.062	0.082	0.082	0.112	0.112
8.000– 9.999	0.054	0.054	0.054		0.064	0.064	0.074	0.074	0.100	0.100	0.136	0.136
10.000–11.999	0.064	0.064	0.064		0.074	0.074	0.088	0.088	0.116	0.116	0.160	0.160
12.000–13.999	0.074	0.074	0.074		0.084	0.084	0.100	0.100	0.134	0.134	0.184	0.184
14.000–14.999	0.080	0.080	0.080		0.090	0.090	0.106	0.106	0.142	0.142	0.196	0.196

[a] The tolerance applicable to a dimension composed of two or more component dimensions is the sum of the tolerances of the component dimensions if all of the component dimensions are indicated.

At points less than ¼ in. from base of leg, the tolerances shown in column 2A are applicable.

[c] Where the space is completely enclosed (hollow shapes), the tolerances in column 2 are applicable for dimensions that can be taken completely over solid metal.

[d] Where the dimensions specified are outside and inside, rather than the wall thickness itself, tolerance on wall thickness shall be plus or minus 10 per cent of mean wall thickness, maximum plus or minus 0.060, minimum plus or minus 0.010.

[e] Tolerance to be as agreed upon between customer and producer.

[f] Where projecting elements involved are less than 0.250 in. thick and have a width-thickness ratio greater than 8, use the B column in columns 4 through 7; otherwise, use the A columns.

[g] Intermediate dimensions shall be rounded off to the third decimal place in accordance with the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29) 1952 Book of ASTM Standards, Part 2.

[h] W and T are defined as the width and thickness respectively of an element of a section. Fig. 1 shows examples of W and T for various elements.

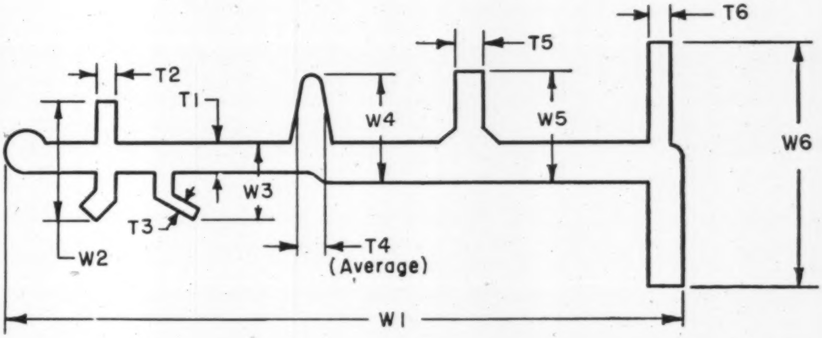


FIG. 1.

TABLE VI.

Alloy and Temper	Form	Cross Sectional Area sq. in.	Compressive Yield Strength min., psi.
ZK60A-F	Bars, Rods, and Shapes	Less than 2.0	27 000
		2.0 to 2.99	26 000
		3.0 to 4.99	25 000
	Hollow Shapes	All	20 000
ZK60A-T5	Bars, Rods, and Shapes	Less than 2.0	30 000
		2.0 to 2.99	28 000
		3.0 to 4.99	25 000
	Hollow Shapes	All	26 000

TABLE VII.

Alloy and Temper	Yield Strength (0.2 per cent offset), min., psi.	Unit Deformation, in. per in. of gage length
ZK60A-F	31 000	0.0068
	28 000	0.0063
ZK60A-T5	36 000	0.0075
	38 000	0.0078

"19,500 min" to read "20,000 min" psi. Delete the last sentence in note *f*.

Specifications for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 52 T):

Table I.—For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point.

Table II.—For alloy M1A-H14 revise the tensile strength requirement of "19,500 min" to read "20,000 min" psi.

Specifications for Aluminum and Aluminum-Alloy Bars, Rods and Wire (B 211 – 52 T):

Table I.—For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point.

Specifications for Magnesium-Base Alloy Extruded Round Tubes (B 217 – 49 T):

Section 1.—Revise to read as follows:

1. These specifications cover commercial magnesium-base alloy extruded tubes of the compositions given in Table I.[3]

Table I.—Delete note *a* and alloy AZ80A and all requirements pertaining to it. Add requirements for alloy ZK60A as follows:

Magnesium, per cent	remainder
Zinc, per cent	4.8 to 6.2
Zirconium, min., per cent	0.45
Other Impurities, max., per cent	0.3

TABLE VIII.—CHEMICAL REQUIREMENTS.

Alloy[a,g]		Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Other Elements, per cent	
											Each	Total
990A		99.0 min.[b]	0.20	[c]	[c]	0.10	...	0.10	...	...	0.05	0.15
996A		99.6 min.[b]	0.05	[d]	[d]	...	...	...	...	...	0.05	0.15
G1A		remainder	0.25	0.8	0.50	0.15	1.0 to 1.8	0.25	0.10	...	0.05	0.15
GR20A		remainder	0.10	[e]	[e]	0.10	2.2 to 2.8	0.20	0.15 to 0.35	...	0.05	0.15
GR40A		remainder	0.10	[e]	[e]	0.10	3.1 to 3.9	0.20	0.15 to 0.35	0.20	0.05	0.15
GS11A		remainder	0.15 to 0.40	0.7	0.40 to 0.8	0.15	0.8 to 1.2	0.20	0.15 to 0.35	0.15	0.05	0.15
Clad GS11A	Core	remainder	0.15 to 0.40	0.7	0.40 to 0.8	0.15	0.8 to 1.2	0.20	0.15 to 0.35	0.15	0.05	0.15
	Cladding	remainder	0.10	[f]	[f]	0.10	...	0.8 to 1.3	...	...	0.05	0.15
M1A		remainder	0.20	0.7	0.6	1.0 to 1.5	...	0.10	...	...	0.05	0.15
Clad M1A	Core	remainder	0.20	0.7	0.6	1.0 to 1.5	...	0.10	...	...	0.05	0.15
	Cladding	remainder	0.10	[f]	[f]	0.10	...	0.8 to 1.3	...	...	0.05	0.15
MG11A		remainder	0.20	0.7	0.30	1.0 to 1.5	0.8 to 1.3	0.10	...	...	0.05	0.15
Clad MG11A	Core	remainder	0.20	0.7	0.30	1.0 to 1.5	0.8 to 1.3	0.10	...	...	0.05	0.15
	Cladding	remainder	0.10	[f]	[f]	0.10	...	0.8 to 1.3	...	...	0.05	0.15

Table II.—Delete alloy AZ80A and all requirements pertaining to it. Add requirements for alloy ZK60A as shown in the accompanying Table X.

Table V.—Add alloy ZK60A as shown in the accompanying Table XI.

Specifications for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221–52 T):

Table I.—For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point.

Specifications for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234–50 T):

Table I.—For the coating of alloy Clad M1A revise the zinc range of "0.75 to 1.25" to read "0.8 to 1.3" per cent. For all alloys revise the limits in the table and notes to express values of 0.50 and less to two digits to the right of

TABLE IX.—TENSILE REQUIREMENTS.

Temper	Thickness, in.	Tensile Strength, min., psi	Yield Strength,[b] min., psi	Elongation in 2 in., min., per cent
		ALLOY GR40A		
O	0.051 to 3.000	30 000	11 000	18
H32	0.051 to 2.000	36 000	26 000	8
H34	0.051 to 1.000	39 000	29 000	6
H112	0.250 to 3.000	30 000	11 000	8

TABLE X.—TENSILE REQUIREMENTS.

Alloy and Temper	Wall thickness, in.	Tensile Strength, min., psi.	Yield Strength[a] (0.2 per cent offset), min., psi.	Elongation in 2 in., min., per cent
ZK60A-F[c]	0.050 to 0.250[b]	40 000	28 000	5
ZK60A-T5[c]	0.050 to 0.250[b]	46 000	38 000	4

TABLE XI.

Alloy and Temper	Yield Strength (0.2 per cent offset), min., psi.	Unit Deformation, in. per in. of gage length
ZK60A-F	28 000	0.0063
ZK60A-T5	38 000	0.0078

the decimal point, and values of greater than 0.50 to one digit to the right of the decimal point.

Section 6.—Revise minimum tensile strength requirement of "19,500" to read "20,000" psi.

Section 11.—Revise to read: "Each length of tubing 1½ in. or less in diameter shall be subjected to an internal air pressure of 250 psi for 5 sec while immersed in water or other suitable liquid. Any evidence of leakage shall be cause for rejection."

TABLE XII.—TENSILE REQUIREMENTS.

Alloy	Temper	Thickness, in.	Tensile Strength, min., psi	Yield Strength, min., psi[a]	Elongation in 2 in. or 4× Diameter min., per cent
GR40A....	O or H112	All sizes	30 000	11 000	...

Specifications for Aluminum-Alloy Extruded Tubes (B 235 – 52 T):

Table I.—For alloy GS10A revise the magnesium range of "0.45 to 0.85" to read "0.45 to 0.9" per cent. For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point.

Specifications for Aluminum-Alloy Pipe (B 241 – 50 T):

Table I.—For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point.

Specifications for Aluminum and Aluminum-Alloy Bars, Rods, and Shapes Pressure Vessel Applications (B 273 – 52 T):

Table I.—For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values greater than 0.50 to one digit to the right of the decimal point. Add requirements for alloy GR40A as follows:

Aluminum, per cent............	remainder
Copper, per cent...............	0.10
Iron, per cent..................	c
Silicon, per cent................	c
Manganese, per cent...........	0.10
Magnesium, per cent...........	3.1 to 3.9
Zinc, per cent..................	0.20
Chromium, per cent............	0.15 to 0.35
Titanium, per cent.............	0.20
Other elements, each, per cent....	0.05
Other elements, total, per cent ..	0.15

[c] Iron plus silicon shall not exceed 0.45 per cent.

Table II.—Add requirements for alloy GR40A as shown in accompanying Table XII.

Specifications for Aluminum and Aluminum-Alloy Pipe, and Tube for Pressure Vessel Applications (B 274 – 52 T):

Section 1.—Revise to read as follows:

> 1. These specifications cover Schedules 5, 10, 40 (standard) and 80 (extra heavy) pipe and round tube, either drawn or extruded, of aluminum and the aluminum alloys designated in Table I.
>
> Note.—Aluminum alloy pipe is generally available only in alloys M1A, GR40A, GS10A, GS11A, and CG42A.

Table I.—For alloy GS10A revise the magnesium range of "0.45 to 0.85" to read "0.45 to 0.9." For the coating of alloy Clad M1A revise the zinc range of "0.75 to 1.25" to read "0.8 to 1.3" per cent. Revise the iron plus silicon limits of "0.60" in footnote *c* to read "0.7" per cent. For all alloys revise the limits to express values of 0.50 and less to two digits to the right of the decimal point, and values of greater than 0.50 to one digit to the right of the decimal point.

TABLE XIII.—TENSILE REQUIREMENTS.

Alloy	Temper	Pipe Size, in.	Tube Wall Thickness, in.	Tensile Strength, min, psi	Yield Strength,[a] min, psi	Elongation in 2 in. or 4× Diameter min, per cent
GR40A	O	All	0.010 to 0.450	30 000	11 000	...
	H34	...	0.010 to 0.450	39 000	29 000	...
	H38	Under 1	0.010 to 0.250	45 000	34 000	...

Add requirements for alloy GR40A as follows:

Aluminum, per cent	remainder
Copper, per cent	0.10
Iron, per cent	[d]
Silicon, per cent	[d]
Manganese, per cent	0.10
Magnesium, per cent	3.1 to 3.9
Zinc, per cent	0.20
Titanium, per cent	0.20
Chromium, per cent	0.15 to 0.35
Other elements, each, per cent	0.05
Other elements, total, per cent	0.15

[d] Iron plus silicon shall not exceed 0.45 per cent.

Table II.—Add requirements for alloy GR40A as shown in accompanying Table XIII.

Table III.—Add requirements for ASA Schedules 5 and 10 as shown in the accompanying Table XIV.

REVISION OF TENTATIVE RECOMMENDED PRACTICE

The committee recommends that the following tentative recommended practice be revised as indicated below and continued as tentative:

TABLE XIV.—NOMINAL DIMENSIONS AND WEIGHTS OF ALUMINUM-ALLOY PIPE.

Size, in.	Outside Diameter, in.	ASA Schedule 5		ASA Schedule 10	
		Wall Thickness, in.	Weight, lb per ft[a]	Wall Thickness, in.	Weight, lb per ft[a]
1/8	0.405	...	...	0.049	0.064
1/4	0.540	...	...	0.065	0.114
3/8	0.675	...	...	0.065	0.146
1/2	0.840	0.065	0.186	0.083	0.232
3/4	1.050	0.065	0.237	0.083	0.297
1	1.315	0.065	0.300	0.109	0.486
1 1/4	1.660	0.065	0.383	0.109	0.625
1 1/2	1.900	0.065	0.441	0.109	0.721
2	2.375	0.065	0.555	0.109	0.913
2 1/2	2.875	0.083	0.856	0.120	1.221
3	3.500	0.083	1.048	0.120	1.498
3 1/2	4.000	0.083	1.201	0.120	1.720
4	4.500	0.083	1.354	0.120	1.912
5	5.563	0.109	2.196	0.134	2.688
6	6.625	0.109	2.624	0.134	3.213
8	8.625	0.109	3.429	0.148	4.635
10	10.750	0.134	5.256	0.165	6.453
12	12.750	0.165	7.672	0.180	8.359

Alloy	Conversion Factor
GR40A	0.98

Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 52 T):

Table I.—Add "Y-Antimony" between "T-Tin" and "Z-Zinc."

APPENDIX

ATMOSPHERIC EXPOSURE OF LIGHT METALS

By L. H. Adam[1]

Some of the answers to various corrosion problems in connection with light metals, such as aluminum and magnesium alloys, may be better known ten years from now when ASTM Committee B-7's Atmospheric Exposure Test is completed. It will take this time for the metals to reveal their loss in strength. In some cases, the material may even at first increase in strength due to the natural aging of the alloy.

In 1941, an atmospheric exposure study was completed for five wrought aluminum alloys exposed for ten years at various seacoast and industrial stations. An excellent analysis of these data, together with other results from the Aluminum Research Laboratories, was given by E. H. Dix and R. B. Mears[2] at the Pittsburgh Meeting of the Society in 1946. However, new alloys and fabrication techniques were developed during the intervening years since the former program had been started, and many new questions in regard to their performance were arising. Therefore, the members of ASTM Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, and Committee B-7 on Light Metals and Alloys, Cast and Wrought, were canvassed by a questionnaire on the desirability of additional studies with light alloys. Based on the expressed desire of the members responding, preliminary plans were made to conduct a limited study on fabricated specimens. It was realized from past experience that too large a program would not be practical for a committee to handle and too meager a program would not warrant the effort. A course was laid which adhered to the "middle of the road" policy. Three definite items were to be emphasized:

1. A study on the new alloys appearing in the Society's specifications.
2. A study on fabricated specimens used by industry.
3. A study on casting alloys, both sand and permanent mold type.

Atmospheric exposure conditions were to depend on the success of the Advisory Committee on Corrosion in obtaining suitable test sites. A request was made for at least two marine sites, one on the West Coast, the other on the East Coast, as well as the possibilities of industrial and rural exposures. Recently five selected sites of the desired exposure conditions have been established. The industrial site is located on the top of the Port of Authority Building in the center of New York City; the rural atmospheric site is covered by the old "stand-by" location at State College, Pa.; the East Coast site, 800 ft from the ocean, is at Kure Beach, N. C., under the jurisdiction of the International Nickel Co.; the West Coast site is located at Point Reyes, San Francisco, Calif.; and the third seacoast site is located on land under control of the Corps of Engineers in the flood gate area at the mouth of the Brazos River in Freeport, Tex.

Only these standard alloys which were not covered in the previous program of the early 30's and those alloys which appeared in 1947 as likely to be promising in the near future are included in the present program. The 27 aluminum and 8 magnesium alloys selected are shown in Table I. The shape and form of materials to be investigated is also indicated: sheet and plate panels, welded and riveted specimens, as well as sand and permanent mold castings. This was a rather ambitious collection. By full industrial cooperation, test specimens were fabricated and identified. They represented commercial materials. The sheet material was in the form of 0.064 in. thick panels. The plate was 0.25 in. thick, made into the normal 4 by 8-in. exposure panels. In the case of welded and riveted joints, the specimens were fabricated to sizes suitable for study. Samples of these specimens are shown in Figs. 1 to 23. The location of the spot welds and rivets will permit tension specimens to be taken from the exposed panels and the data studied at a later date.

---

[1] Materials Engineer, Pitman-Dunn Laboratory, Frankford Arsenal, Philadelphia, Pa.; Chairman of Subcommittee VIII, ASTM Committee B-7 on Light Metals and Alloys, Cast and Wrought.

[2] "Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals," Am. Soc. Testing Mats. (1946). (Issued as separate publication, *ASTM STP No. 67*.)

TABLE I.—ATMOSPHERIC EXPOSURE TEST—ASTM COMMITTEE B-7.

ASTM Alloy Designation	Commercial Name	Condition	Sheet, 0.064 in. Thick				Plate, 0.25 in. Thick				Extruded Bar ¼ in. by 4 in.			Castings	
			Panel	Spot Welded	Brazed	Riveted	Panel	Gas Welded	Arc Welded	Riveted	Panel	Arc Welded	Riveted	Sand	Permanent Mold
							WROUGHT ALUMINUM								
M1A	3S	H14	X	..	X	..	..	..	..	..	..	..	..	..	..
GR20A	52S	H34	X	..	..	..	..	..	..	..	..	..	..	..	..
GS11A	61S	T6	X	X	X	X	X	X	X	X	X	..	..	..	..
GS10A	63S	T5	..	..	..	..	..	..	..	..	X	X	..	..	..
CS41A	14S	T4	..	..	..	..	X	..	..	X	X	..	..	..	..
CS41A	14S	T6	..	..	..	..	X	..	..	X	X	..	X	..	..
CG42A	24S	T3	X	..	..	X	..	..	..	..	..	..	..	..	..
ZG62A	75S	T6	X	..	..	X	..	..	..	..	X	..	..	..	..
MG11A	4S	H34	X	..	..	..	..	..	..	..	..	..	..	..	..
G1A	50S	H34	X	..	..	..	..	..	..	..	..	..	..	..	..
Clad CS41A	Alclad 14S	T6	X	X	..	X	X	..	..	X	..	..	..	..	..
Clad CG42A	Alclad 24S	T6	X	X	..	X	X	..	..	X	..	..	..	..	..
Clad ZG62A	Alclad 75S	T6	X	X	..	X	X	..	..	..	..	..	..	..	..
							WROUGHT MAGNESIUM								
M1A	M	..	X	X	X	X	..	..	..	..	X	..	..	..	..
AZ31A and B	FS1	..	X	X	..	X	X	..	..	..	X	..	X	..	..
AZ80A	AMC58S	..	..	..	..	..	..	..	..	..	X	..	..	..	..
ZK60A	ZK60	..	..	..	..	..	..	..	..	..	X	..	X	..	..
							ALUMINUM CASTING ALLOYS								
S5B	43	F	..	..	..	..	..	..	..	..	..	..	..	X	X
C4A	195	T6	..	..	..	..	..	..	..	..	..	..	..	X	..
G10A	220	T4	..	..	..	..	..	..	..	..	..	..	..	X	..
SC51A	355	T6	..	..	..	..	..	..	..	..	..	..	..	X	X
SG70A	356	T6	..	..	..	..	..	..	..	..	..	..	..	X	..
SC64B	Allcast	T6	..	..	..	..	..	..	..	..	..	..	..	X	X
ZG61A	40E	T5	..	..	..	..	..	..	..	..	..	..	..	X	..
CS43A	108	F	..	..	..	..	..	..	..	..	..	..	..	X	..
ZG32A	Ternalloy 5	..	..	..	..	..	..	..	..	..	..	..	..	X	X
ZC81	Tenzalloy	T5	..	..	..	..	..	..	..	..	..	..	..	X	..
ZG61B	A612	T5	..	..	..	..	..	..	..	..	..	..	..	X	..
ZC60A	C612	T5	..	..	..	..	..	..	..	..	..	..	..	..	X
ZG81B	Tenzalloy	T5	..	..	..	..	..	..	..	..	..	..	..	..	X
ZG42A	Ternalloy 7	T5	..	..	..	..	..	..	..	..	..	..	..	X	X
							MAGNESIUM ALLOY CASTINGS								
AZ63A	AM265	T4	..	..	..	..	..	..	..	..	..	..	..	X	..
AZ91C	R	..	..	..	..	..	..	..	..	..	..	..	..	X	..
AZ92A	AM260	T6	..	..	..	..	..	..	..	..	..	..	..	X	..
EK30A	EK30	..	..	..	..	..	..	..	..	..	..	..	..	X	..

From the many ways of evaluating the

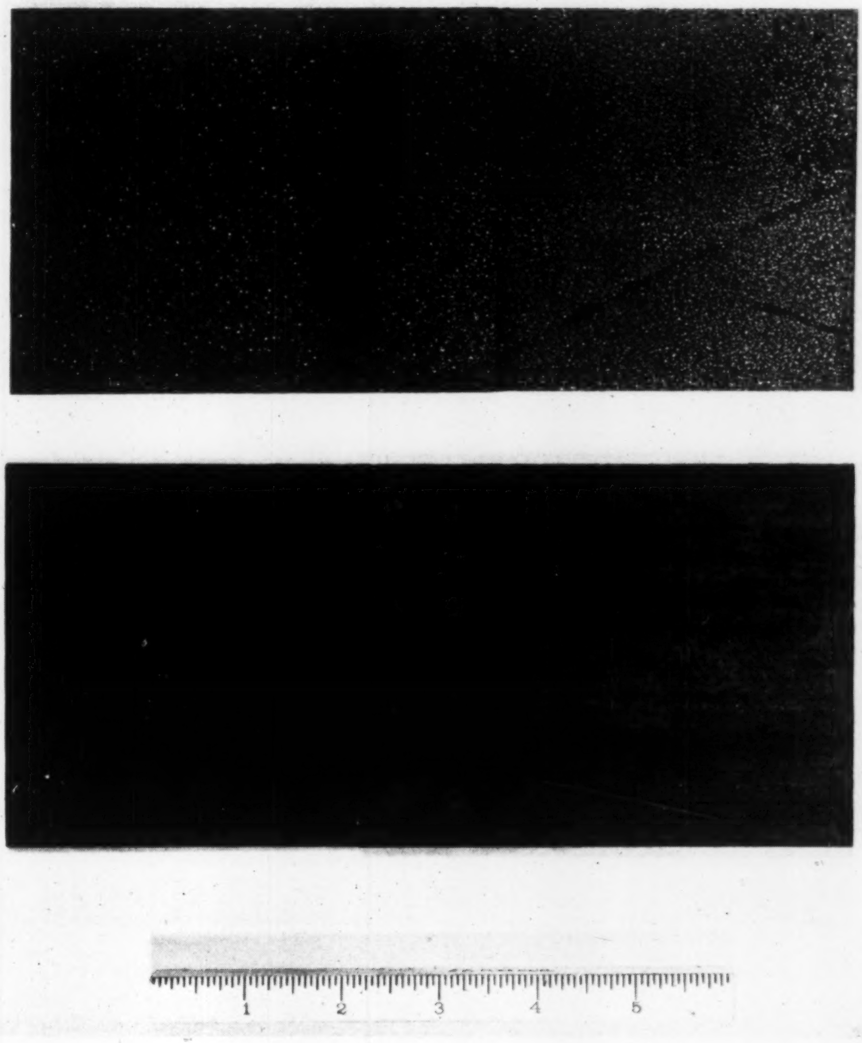


FIG. 1.—Top and Bottom Surfaces of Spot-Welded AZ31A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

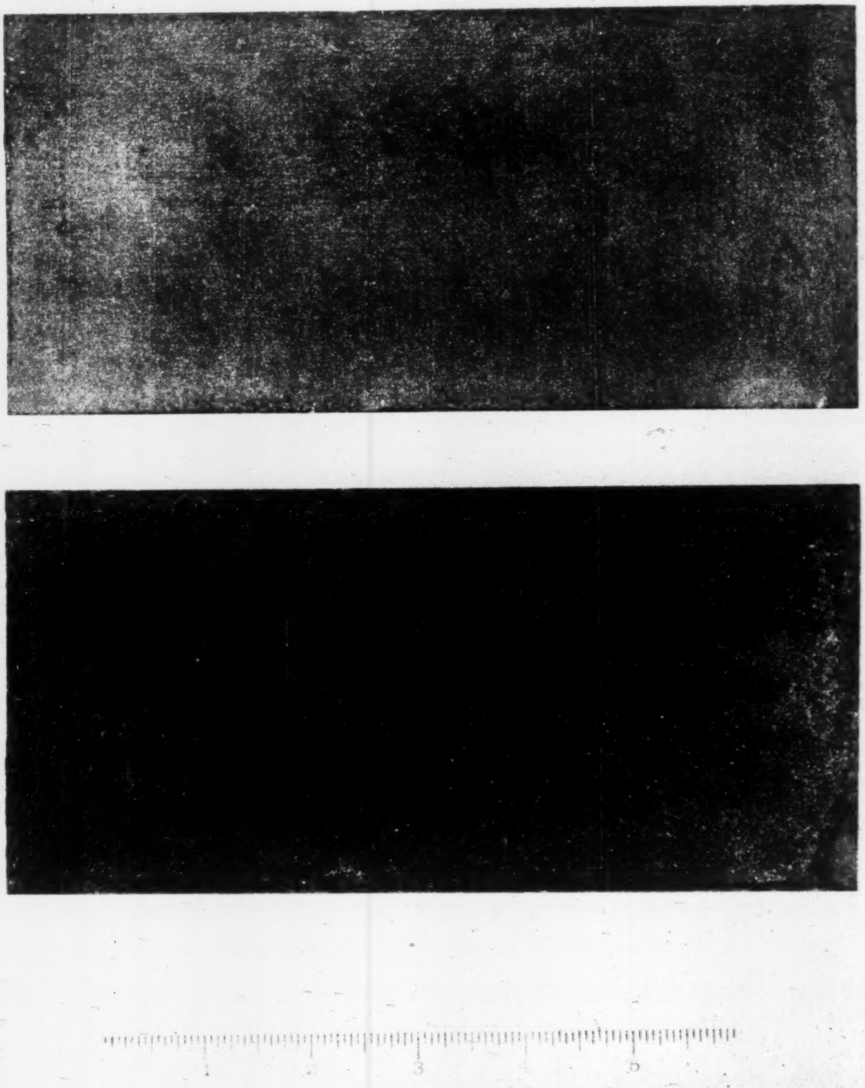


FIG. 2.—Top and Bottom Surfaces of an Extruded ZK60A Magnesium Panel, 0.25 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

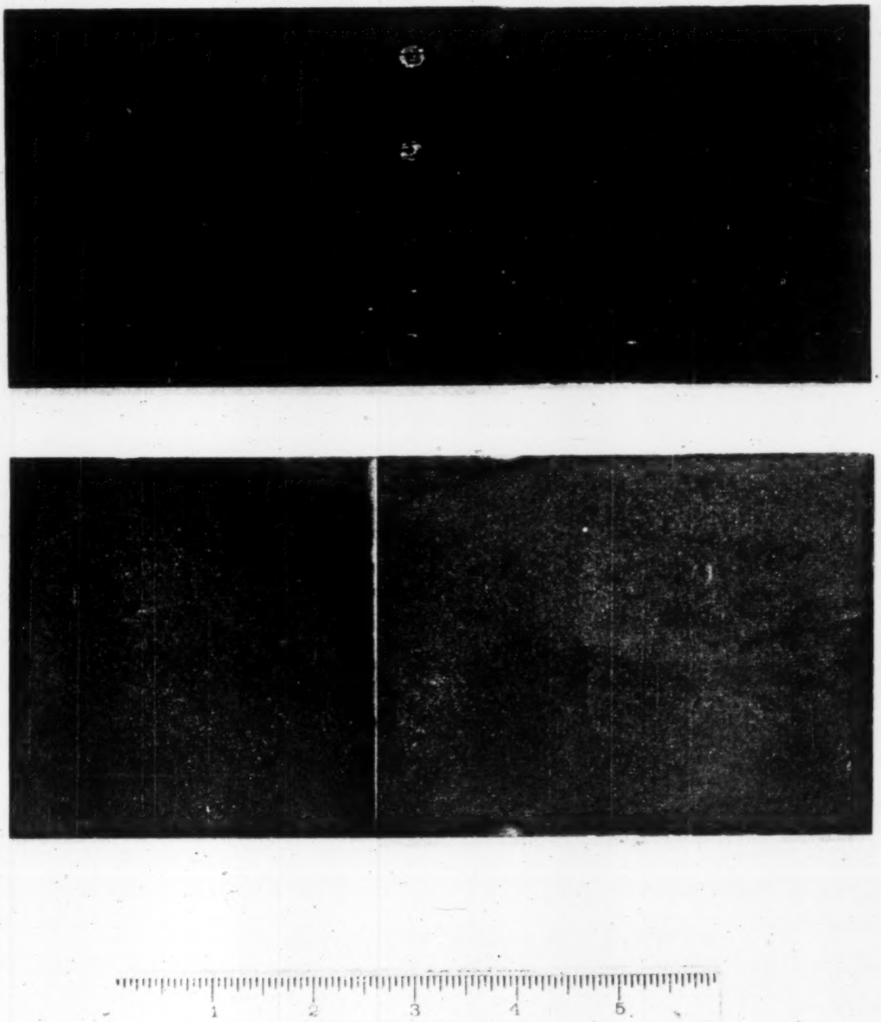


Fig. 3.—Top and Bottom Surfaces of Spot-Welded M1A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

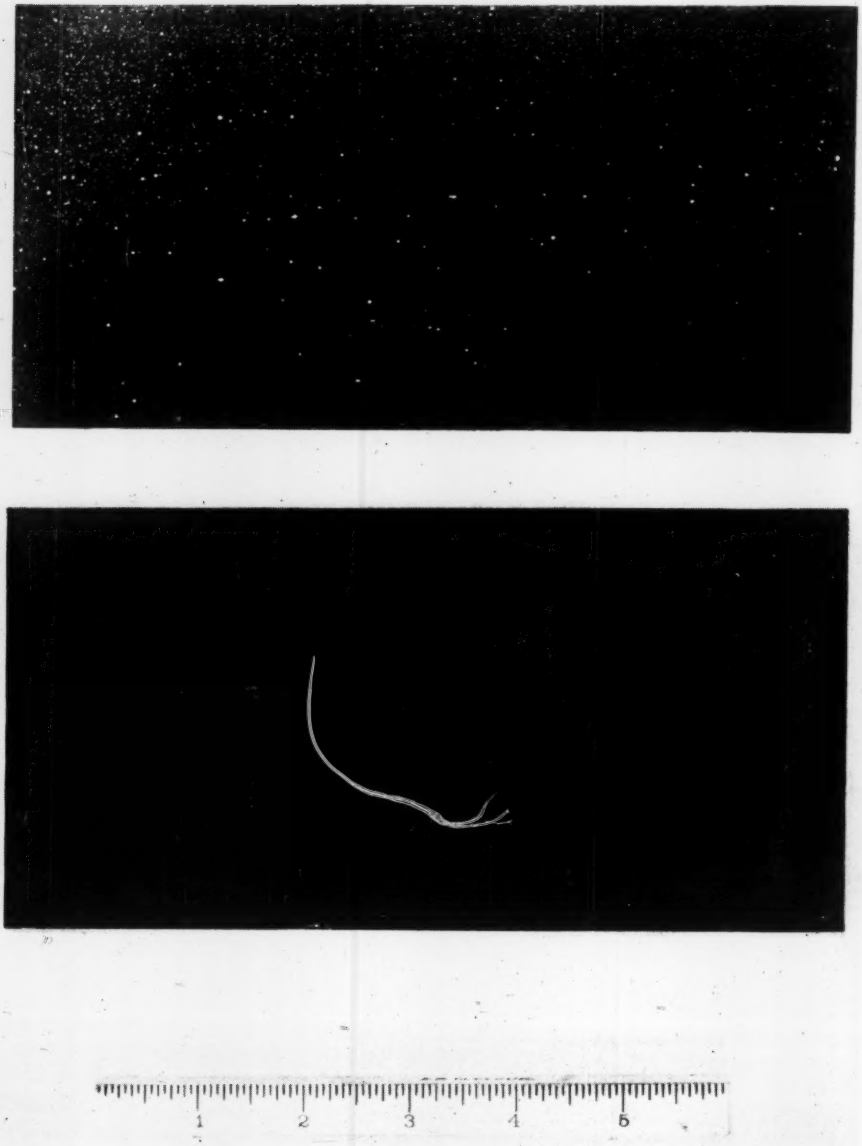


FIG. 4.—Top and Bottom Surfaces of M1A Magnesium Panel, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

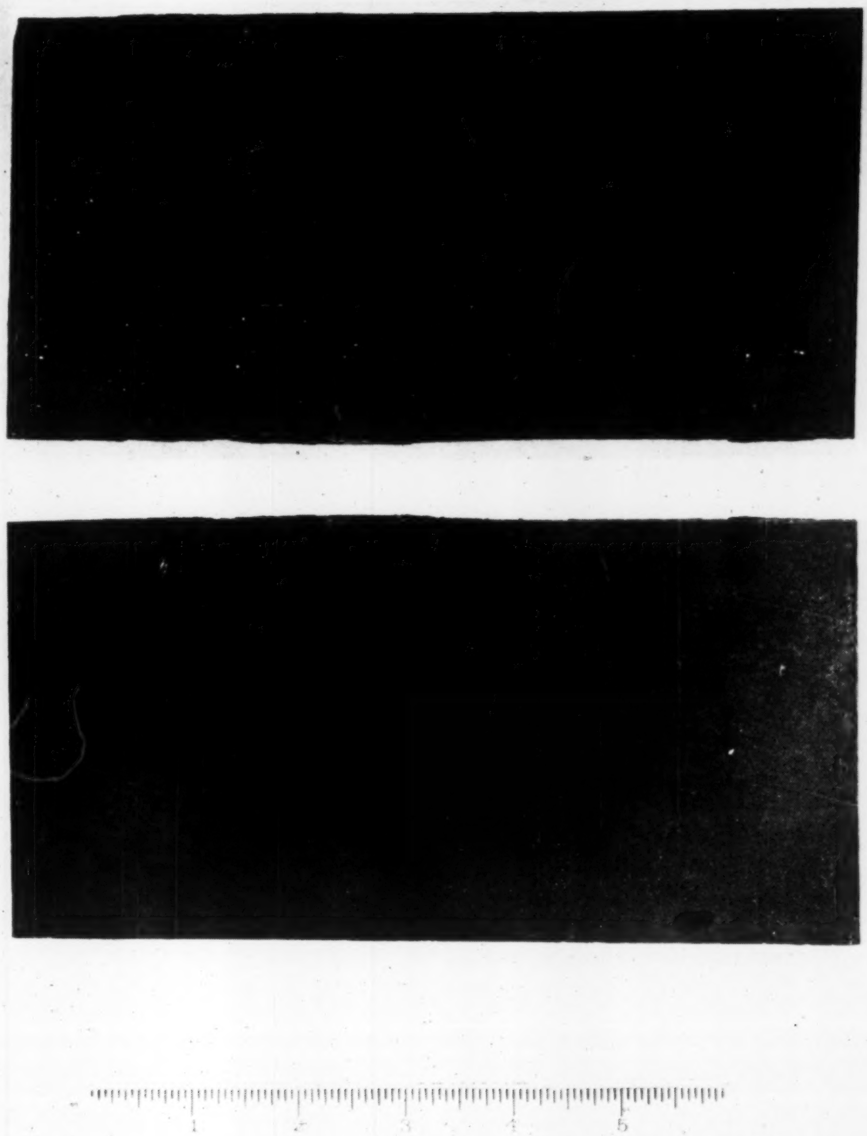


FIG. 5.—Top and Bottom Surfaces of a AZ31A Magnesium Panel, 0.25 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

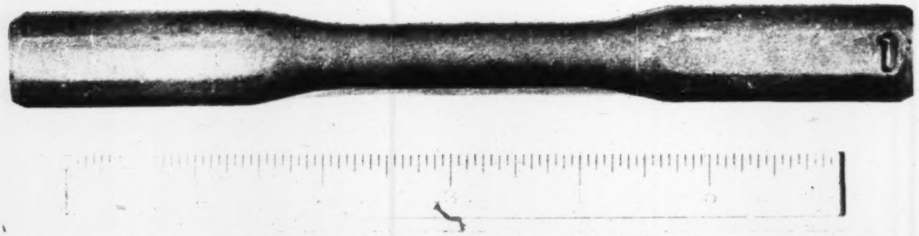


FIG. 6.—Sand-Cast AZ92A Magnesium Tension Specimen, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

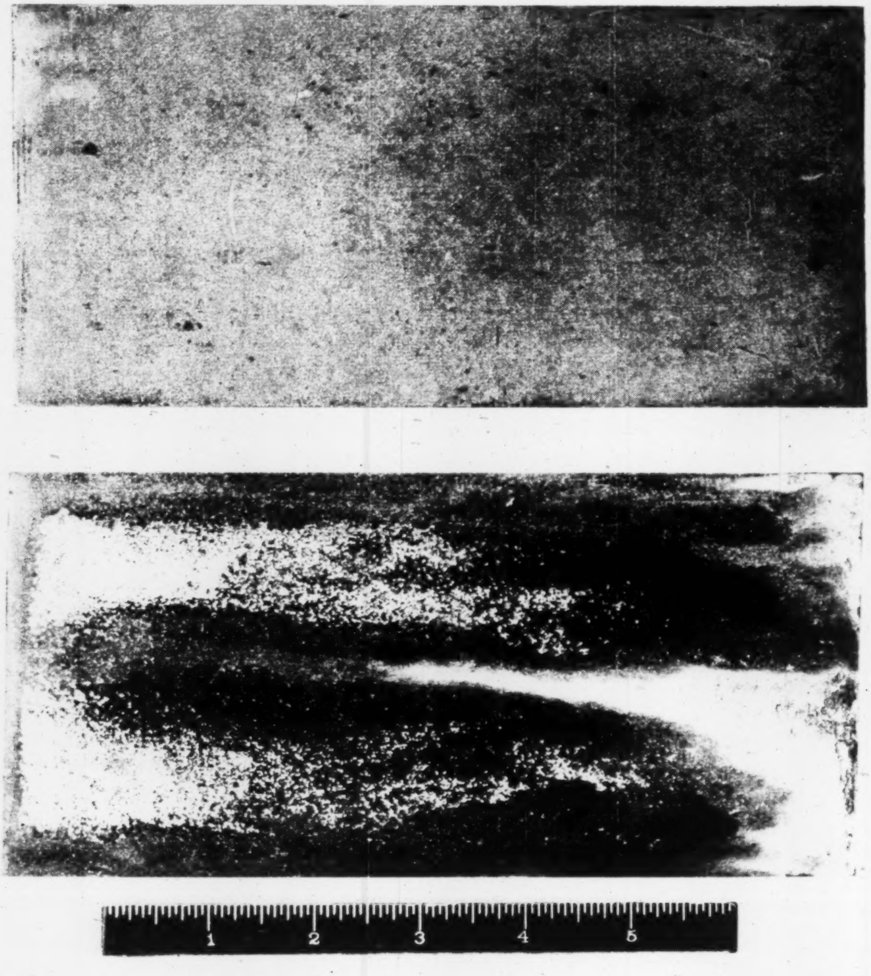


FIG. 7.—Top and Bottom Surfaces of an Extruded ZK60A Magnesium Panel, 0.25 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

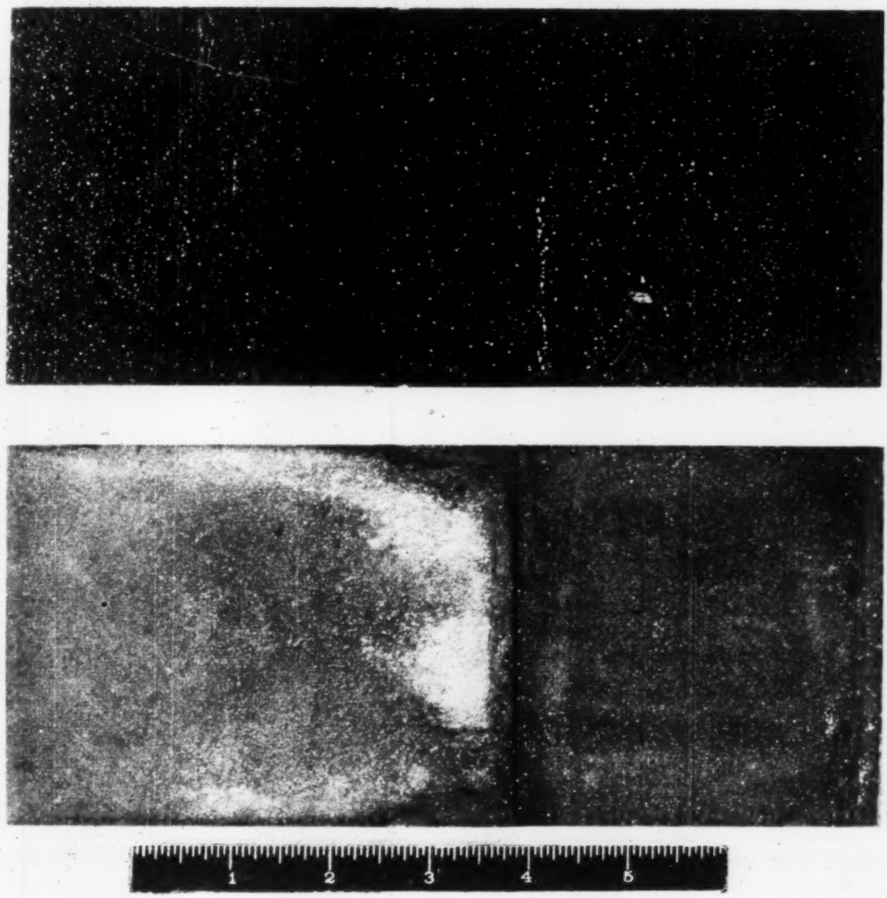


FIG. 8.—Top and Bottom Surfaces of Spot-Welded AZ31A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

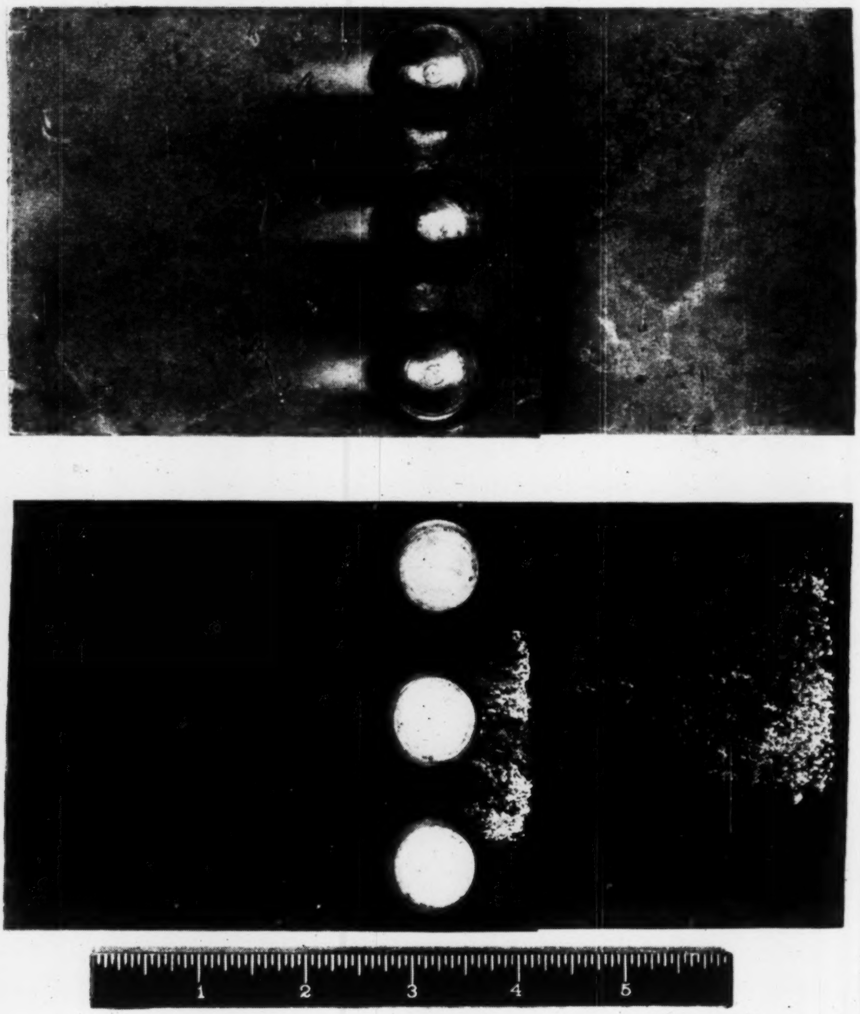


FIG. 9.—Top and Bottom Surfaces of a Riveted ZK60A Magnesium Specimen, 0.25 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

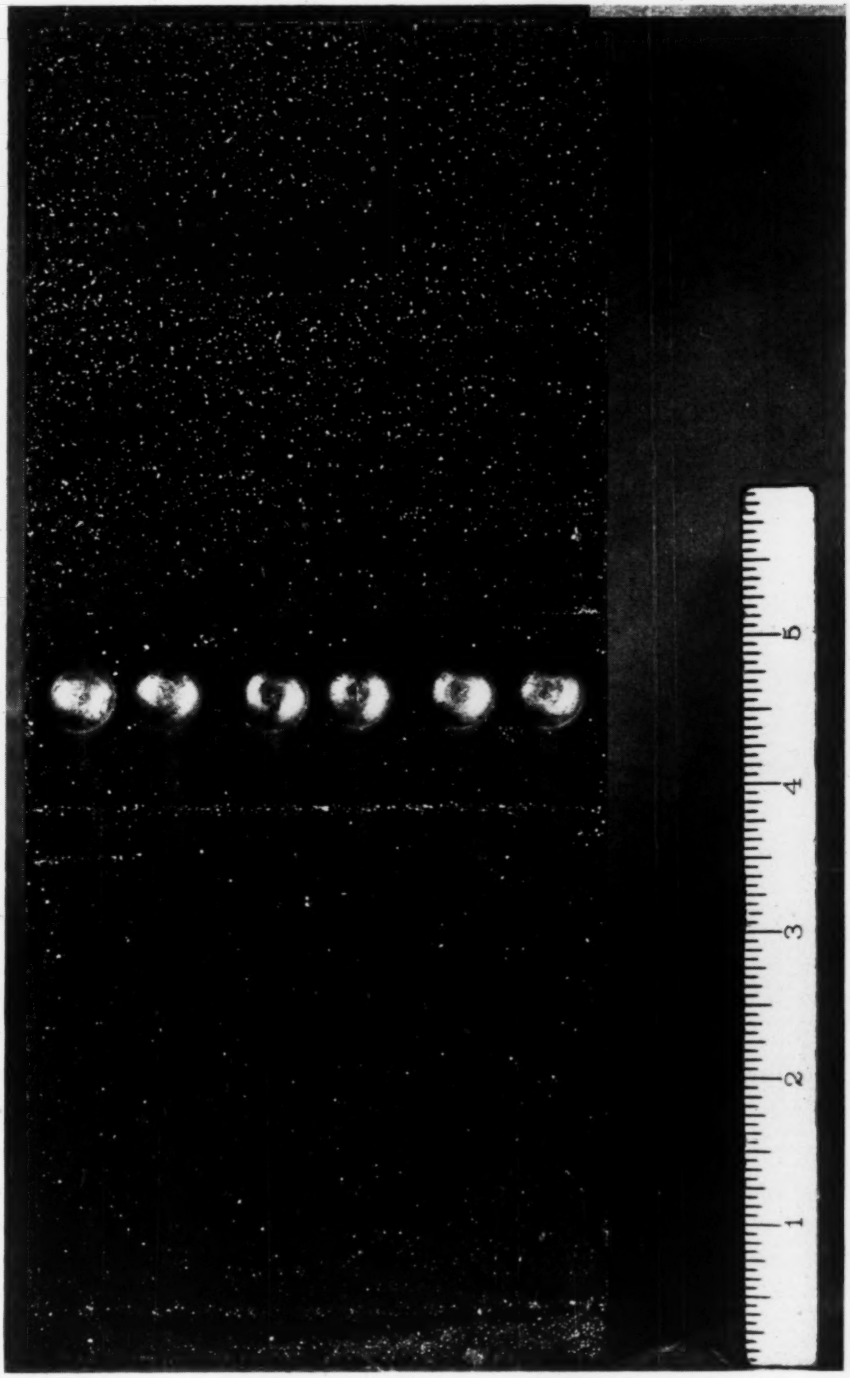


FIG. 10.—Top Surface of a Riveted AZ31A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

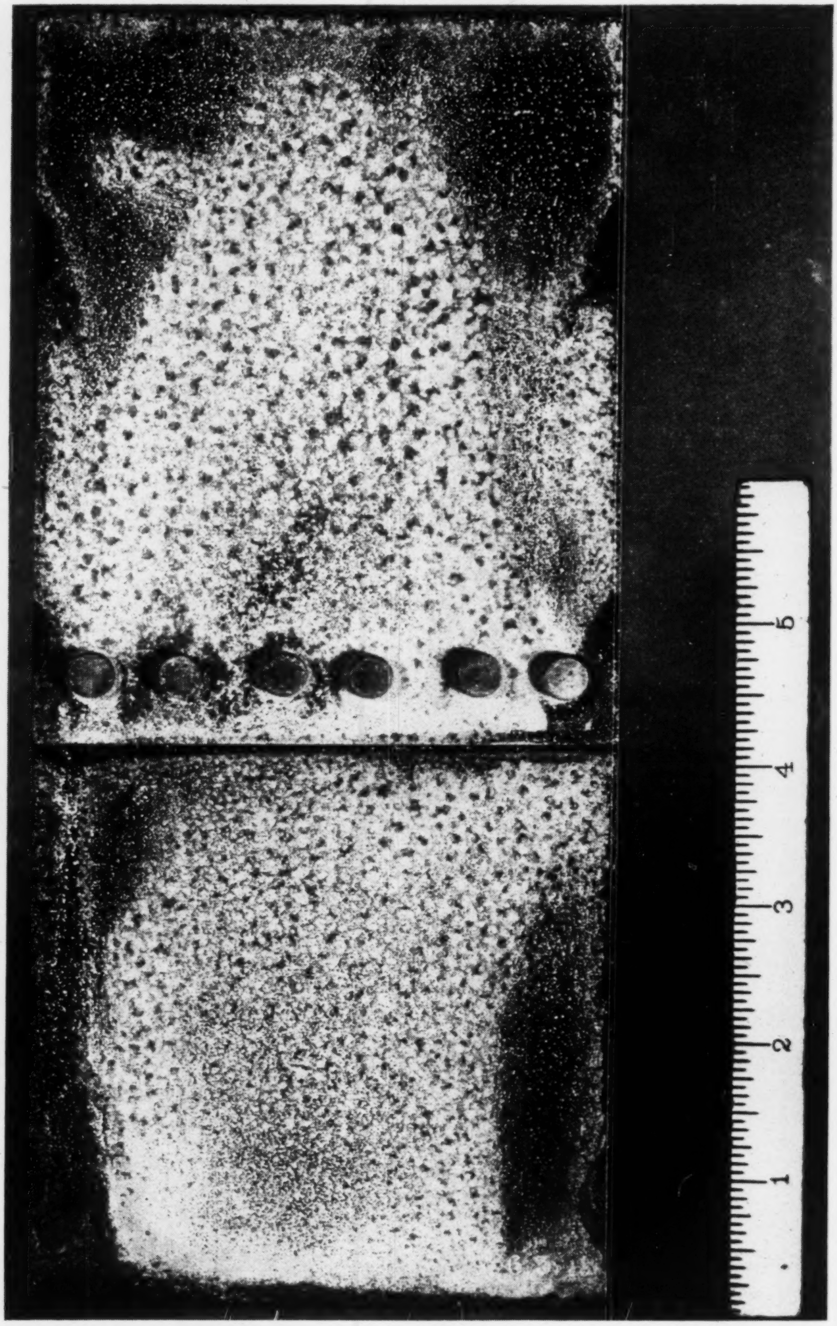


FIG. 11.—Bottom Surface of a Riveted AZ31A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

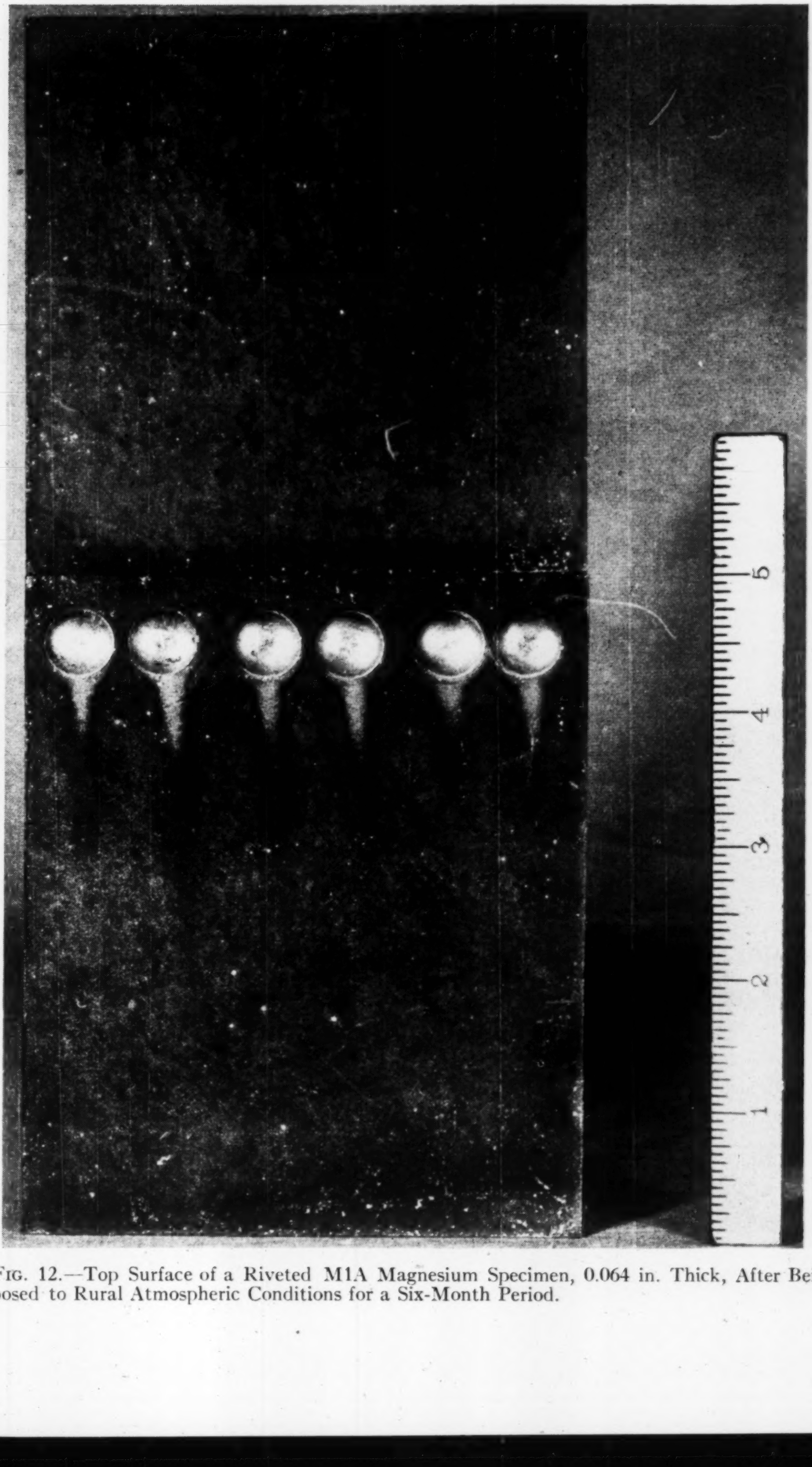


FIG. 12.—Top Surface of a Riveted M1A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

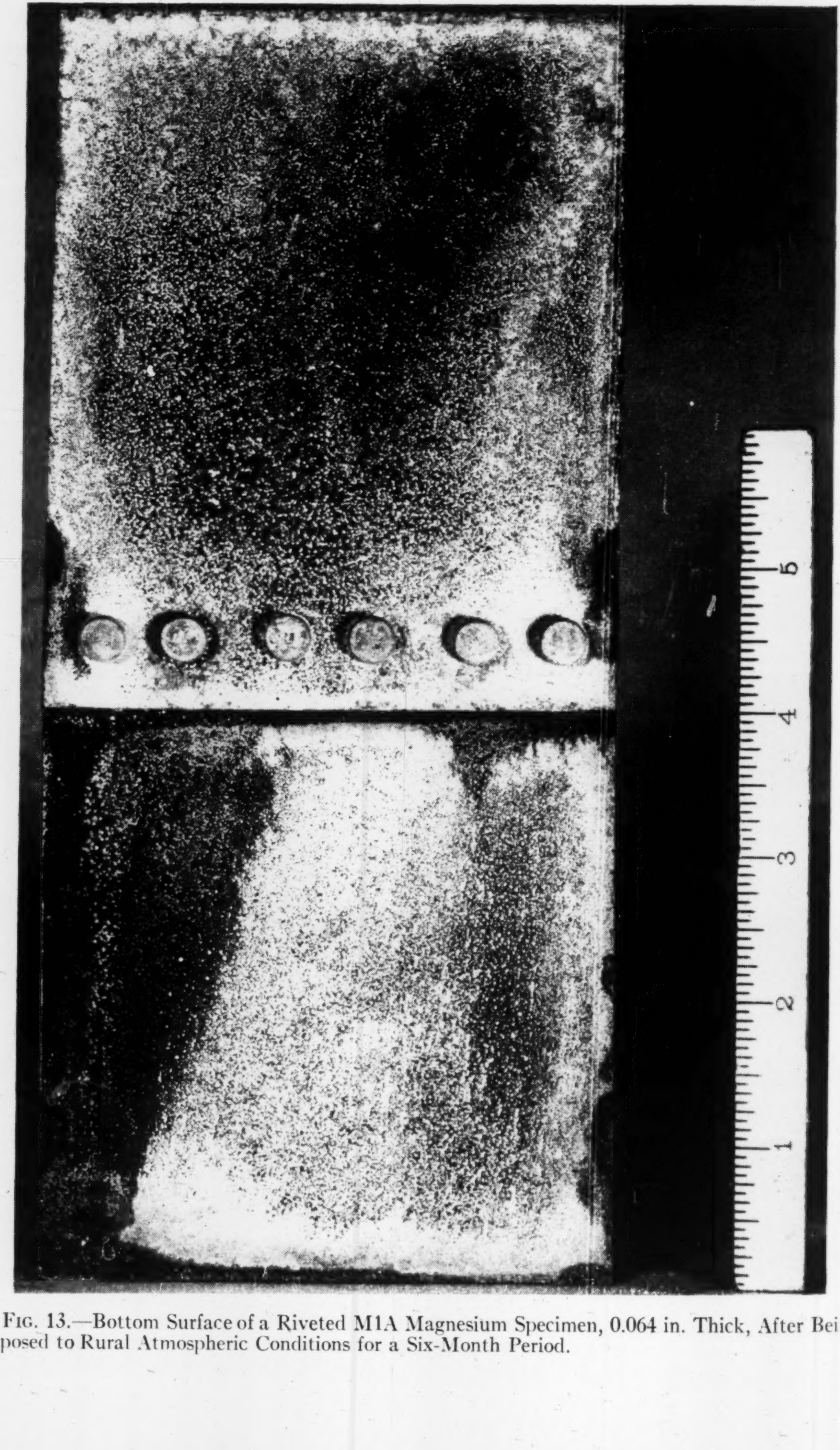


FIG. 13.—Bottom Surface of a Riveted M1A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Six-Month Period.

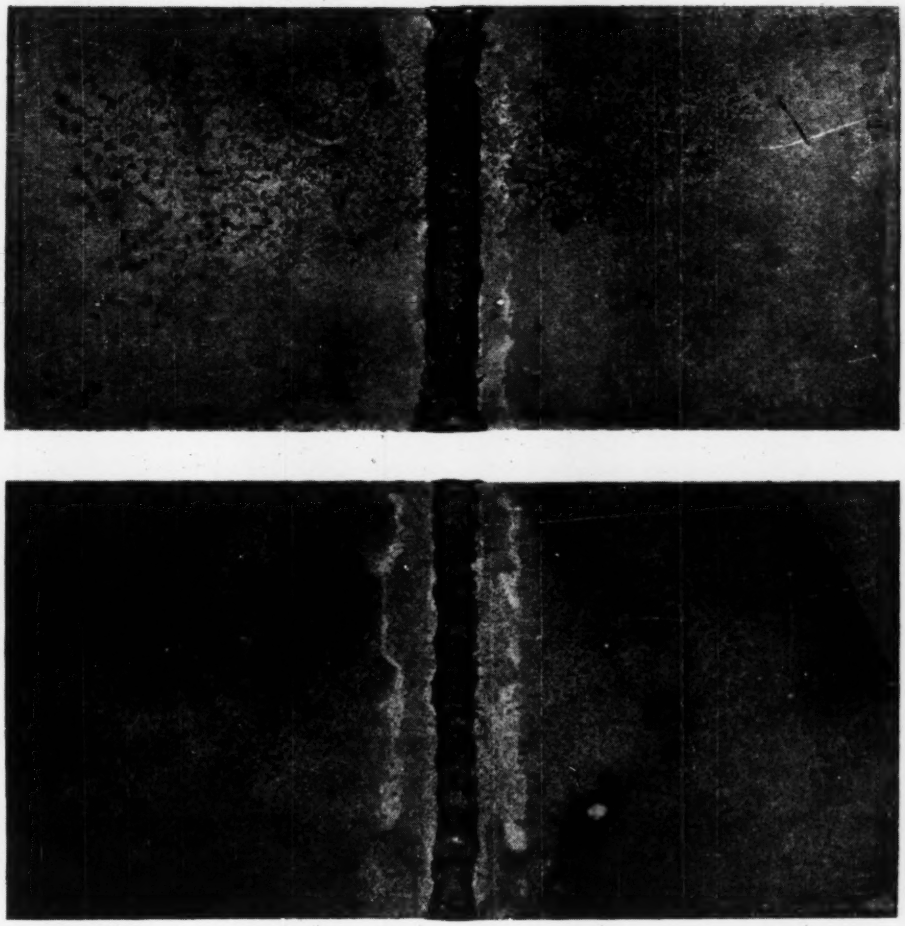


FIG. 14.—Top and Bottom Surfaces of a Gas-Welded GS11A Aluminum Specimen, 0.25 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

effects of exposure, the change in mechanical properties was decided as meeting the needs of the committee. Quoting from Dix and Mears'[2] analysis: "For aluminum alloys, changes in tensile strength resulting from exposure are of more value in determining effects of exposure than are weight changes. Measurement of depth of attack by the depth gages, as was done in the ASTM test, give results considerably less than those obtained by microscopic measurements of cross-sections, hence are of very doubtful value." It was therefore established that all specimens would be properly identified, their dimensions recorded, and then submitted to a random distribution for placement at the exposure sites. For testing for mechanical properties, tension specimens for the wrought materials were to be taken from panels and tested as outlined in the Society's methods E 8.[3] The cast alloys were to comply with Specifications B 26, B 80, or B 108.[4] All the specimens were to be "mill condition." No consideration was to be given to supplemental protective finishes, as at this stage the program was becoming too large for the com-

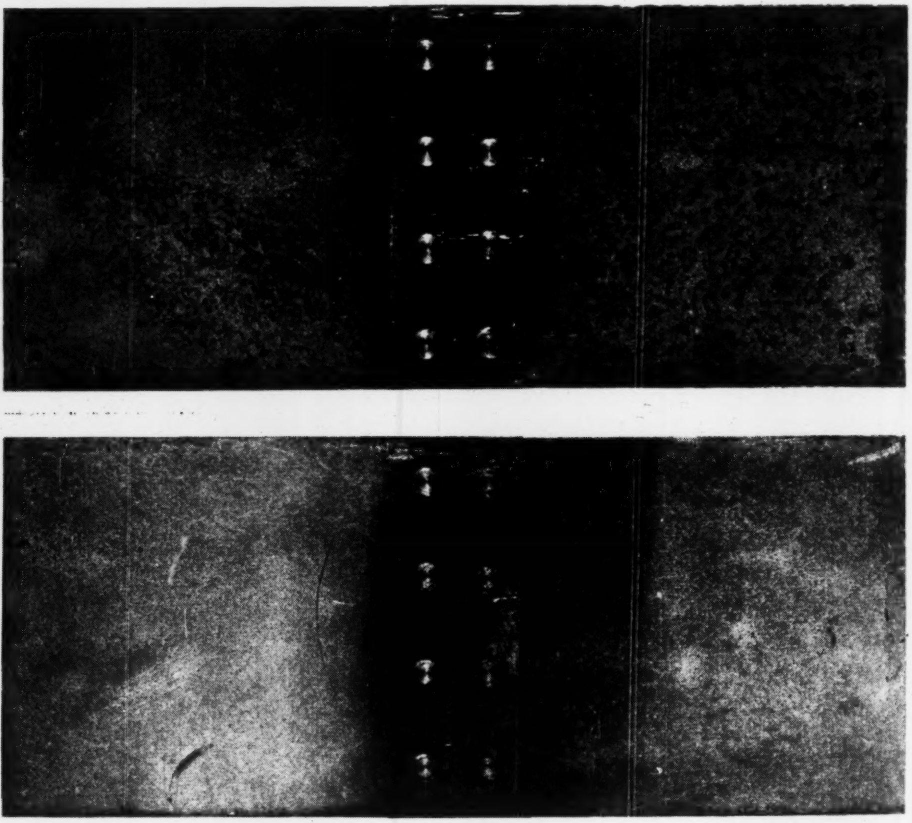


FIG. 15.—Top and Bottom Surfaces of A Spot-Welded CG42A Clad Aluminum Specimen, 0.064 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

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[3] Tentative Methods of Tension Testing of Metallic Materials (Designation: E 8 – 52 T), 1952 Book of ASTM Standards, Part 2.

[4] Specifications for aluminum-alloy sand castings (B 26), magnesium-alloy sand castings (B 80), and aluminum-alloy permanent mold castings (B 108); 1952 Book of ASTM Standards, Part 2.

mittee to handle. Some day, particular phases pertaining to finishes for the light alloys will have to be studied as their individual conditions warrant. At first the program was rather slow in getting started as specimens had to be collected and fabricated under production conditions. Finally, after four years, all specimens were gathered and identified, keeping in mind at all times the previous warning of the statisticians on random distribution. Specimens have now been exposed for one year at the New York and State College sites, and at least six months at the Kure Beach site. Due to delays in negotiating and erecting exposure facilities at the Gulf Coast and West Coast locations, specimens have not been placed in their exposure racks. It is expected that these specimens will be in their proper locations by the middle of 1953.

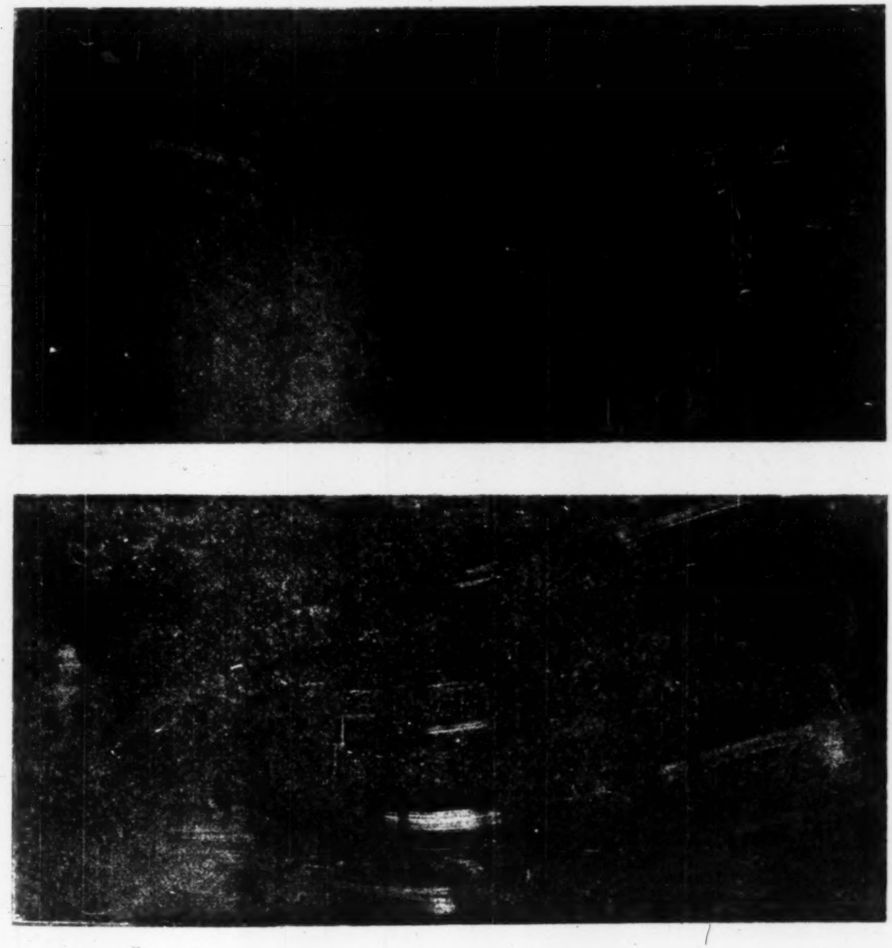

FIG. 16.—Top and Bottom Surfaces of a ZG62A Clad Aluminum Panel, 0.064 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

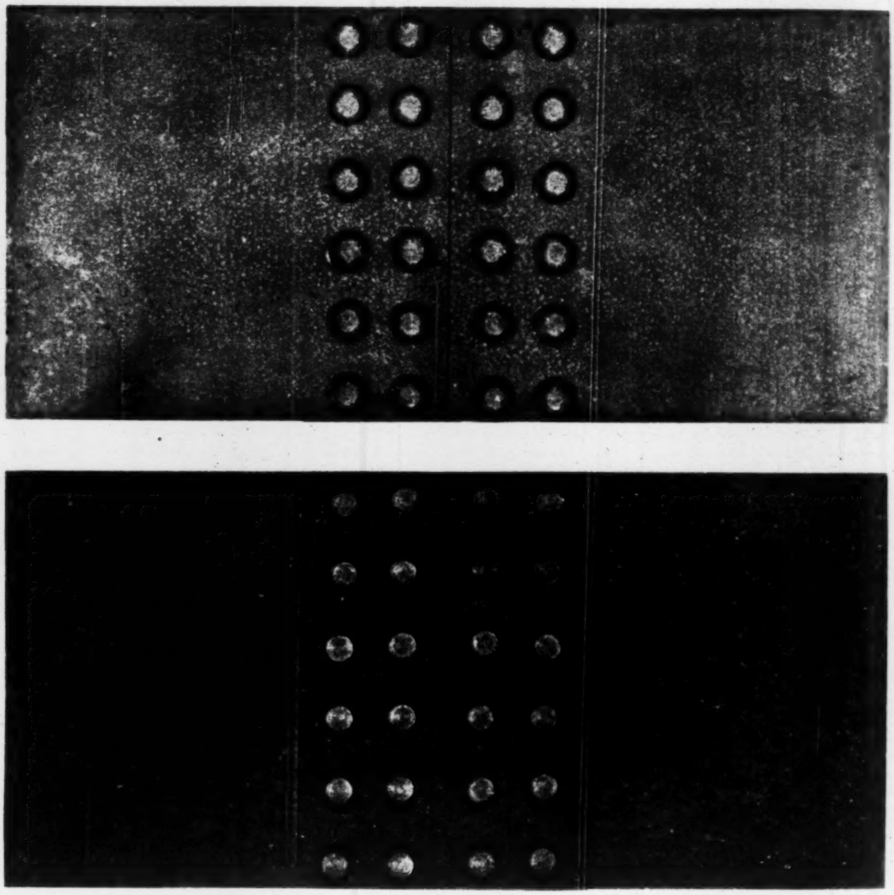

FIG. 17.—Top and Bottom Surfaces of a Riveted ZG62A Aluminum Specimen, 0.064 in. Thick, After Being Exposed to Industrial Atmospheric Conditions for a Six-Month Period.

Periodically, data collected from the exposed specimens are to be reported to Committee B-7 and the data made available for publication. The meager data collected to date are not sufficient to warrant publication.

The program undertaken by Committee B-7 is made possible only by the excellent cooperation by participating members of the Society, and acknowledgment is hereby given to those assisting in the program, particularly staff members of the Pitman-Dunn Laboratories, Frankford Arsenal, where data from the tests are being catalogued and control specimens maintained in sealed containers.

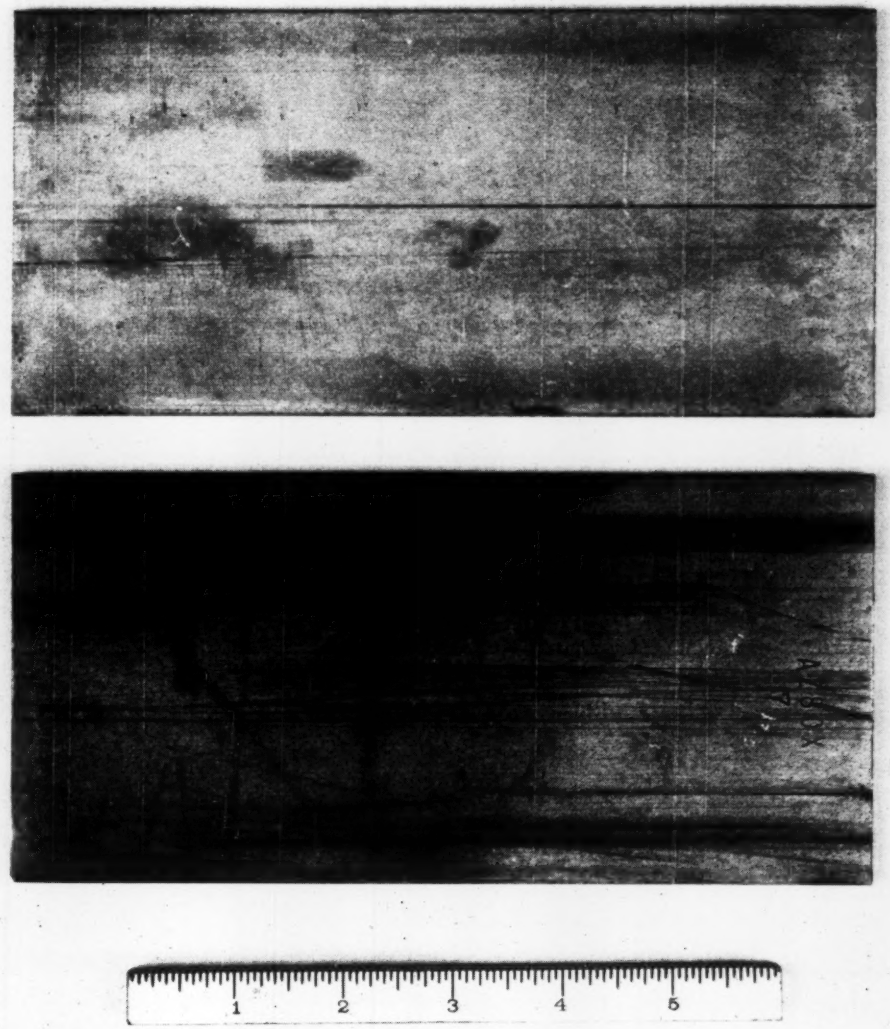

FIG. 18.—Top and Bottom Surfaces of an Extruded AZ80A Aluminum Panel, 0.25 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Period of 1 yr.

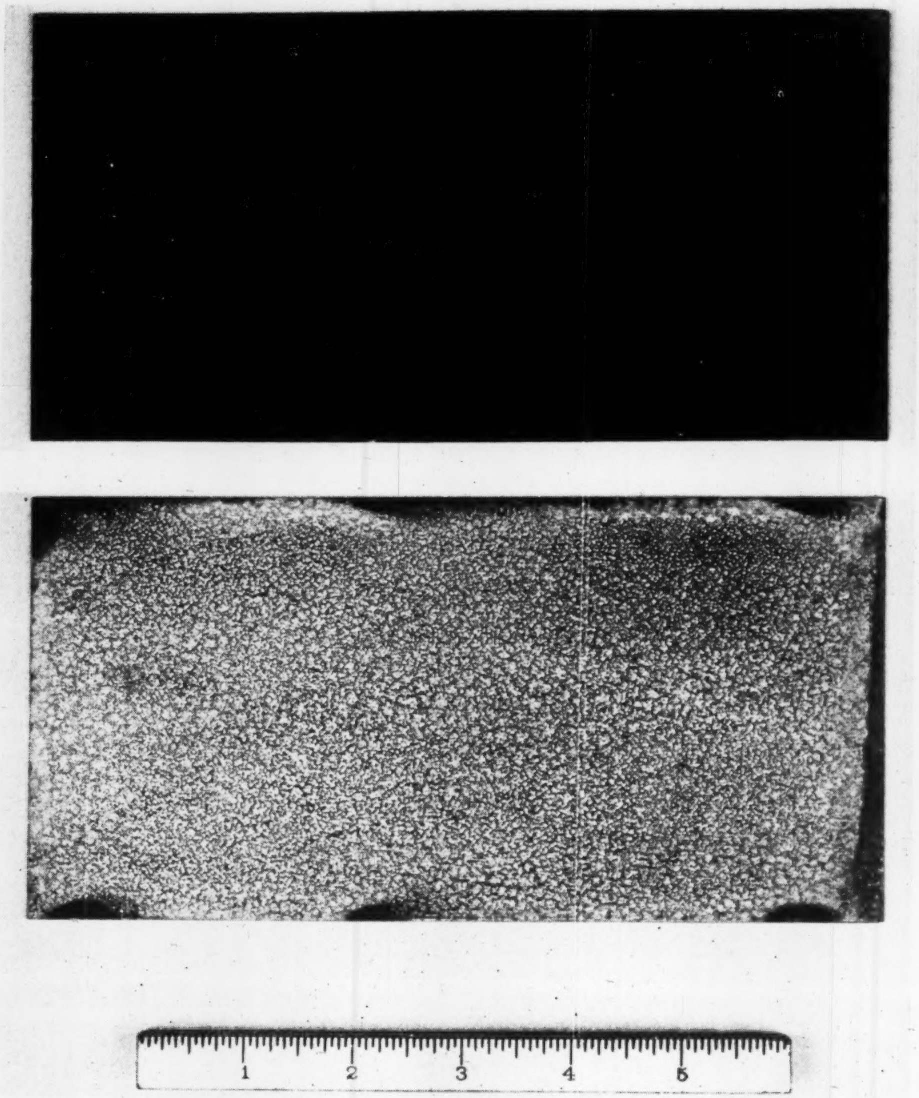

FIG. 19.—Top and Bottom Surfaces of M1A Magnesium Panel, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Period of 1 yr.

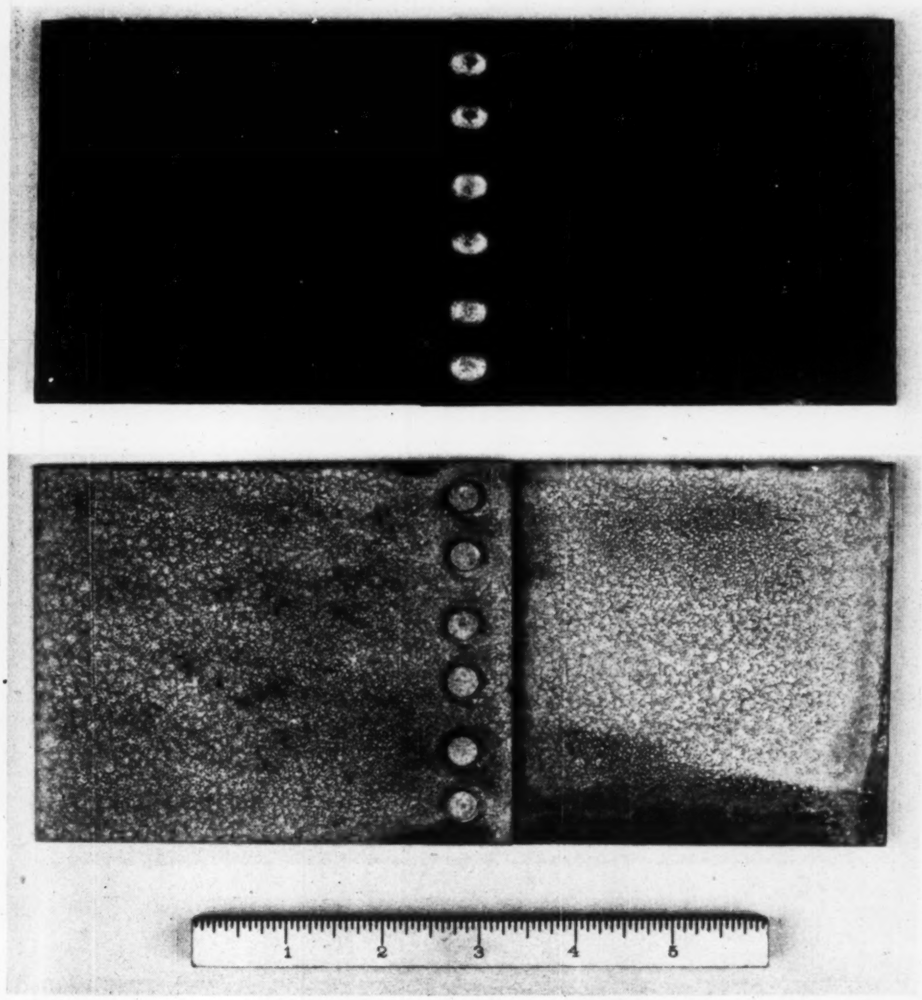

FIG. 20.—Top and Bottom Surfaces of a Riveted M1A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Period of 1 yr.

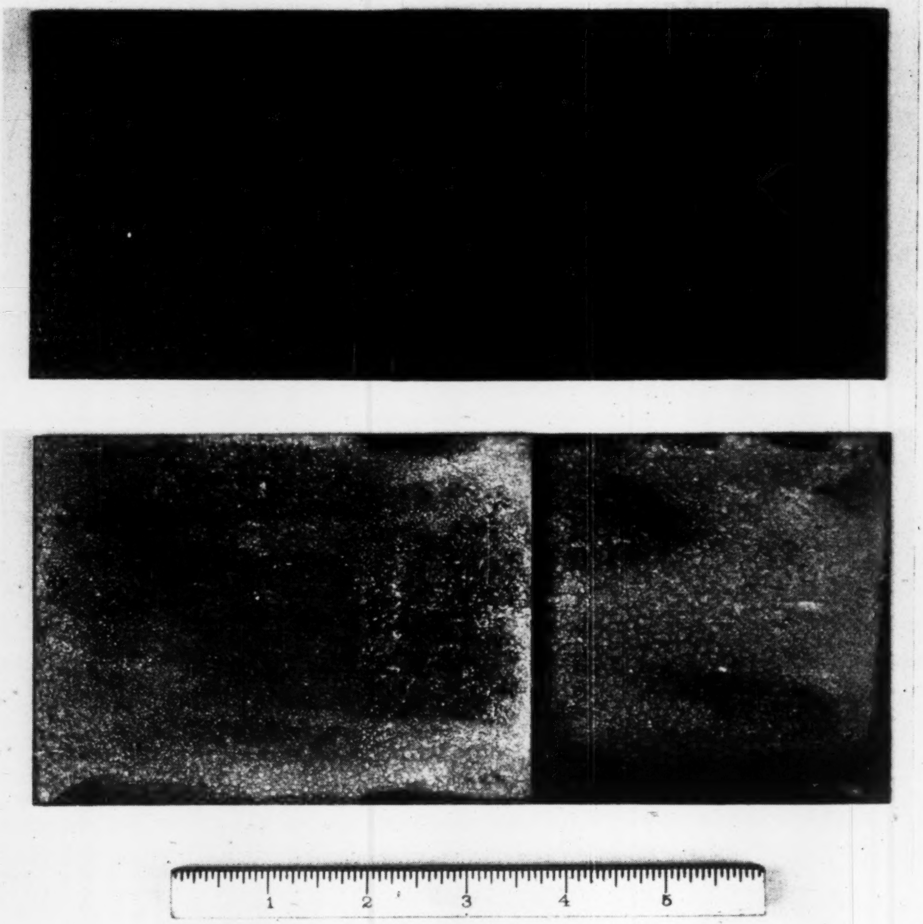

FIG. 21.—Top and Bottom Surfaces of a Spot-Welded AZ31A Magnesium Specimen, 0.064 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Period of 1 yr.

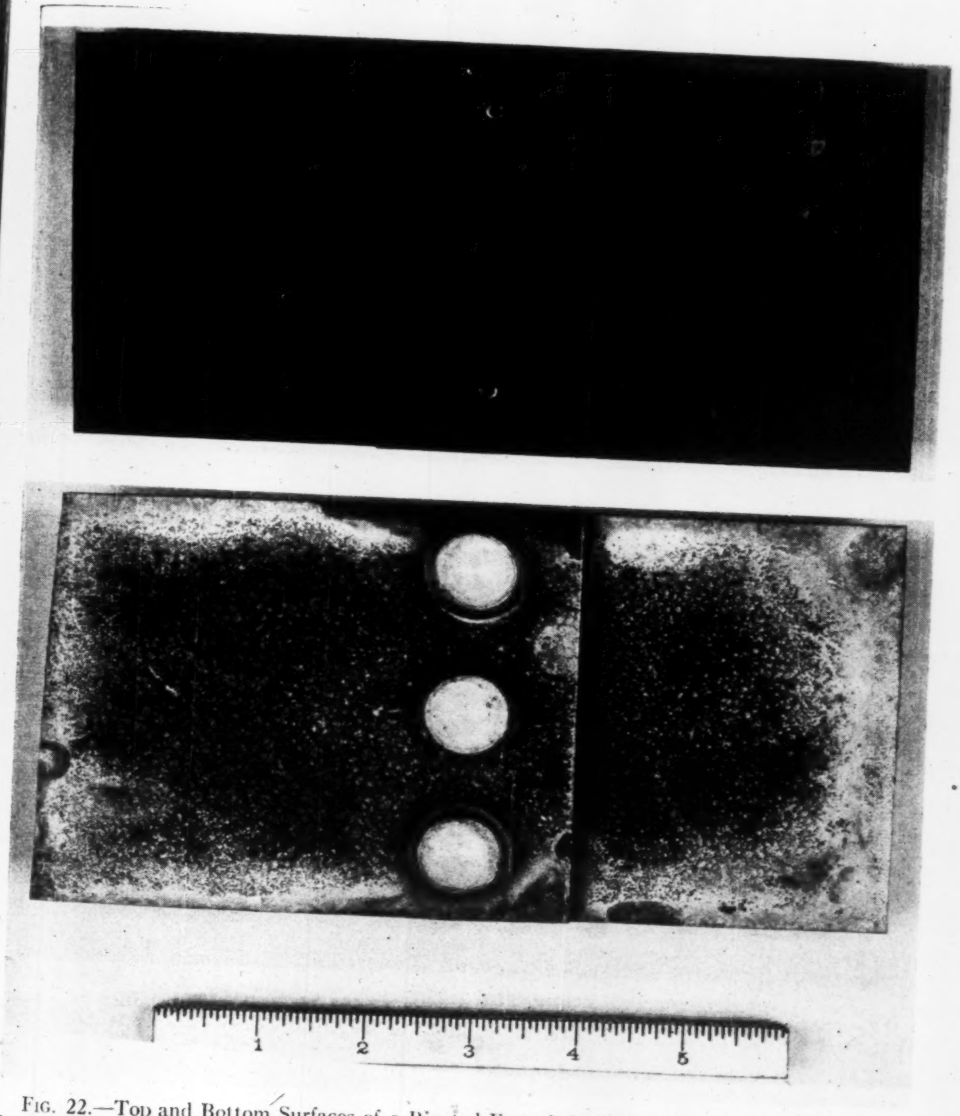

FIG. 22.—Top and Bottom Surfaces of a Riveted Extruded ZK60A Magnesium Specimen, 0.25 in. Thick, After Being Exposed to Rural Atmospheric Conditions for a Period of 1 yr.

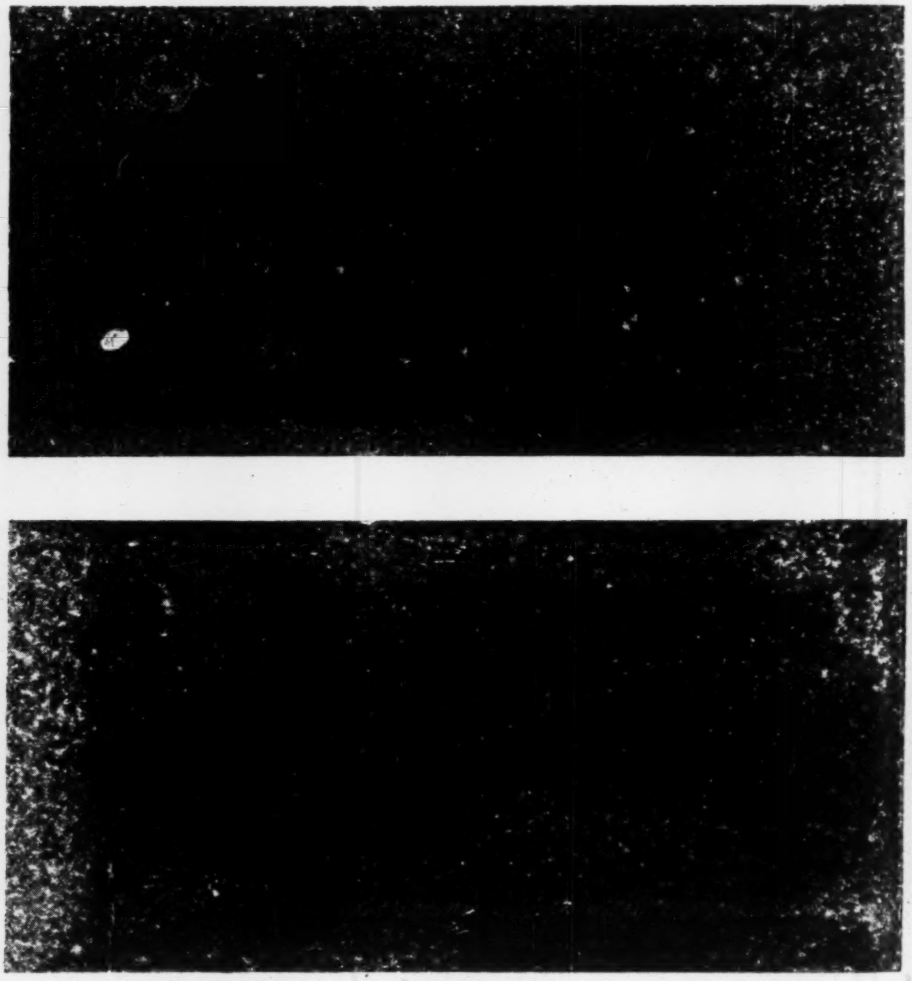

FIG. 23.—Top and Bottom Surfaces of an Extruded ZK60A Magnesium Panel, 0.25 in. Thick, After Being Exposed to Marine Atmospheric Conditions on the Atlantic Coast for a Six-Month Period

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 held two meetings during the year: one on November 14, 1952, at ASTM Headquarters in Philadelphia, Pa., and one in Detroit, Mich., on March 6, 1953.

The membership of the committee now consists of 123 members, of whom 41 are classified as producer, 13 as consumer, 58 as general interest, and 11 as consulting members.

The matter of designating thickness values in mils rather than decimal inches is still under active consideration and is presently being resubmitted to the committee for letter ballot.

NEW TENTATIVE

Committee B-8 recommends that the Tentative Recommended Practice for the Preparation of Copper and Copper-Base Alloys for Electroplating be accepted for publication as tentative, as appended hereto.[1]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following two standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc Base Die Castings (**B 142 - 51**):[2]

Section 2.—Introduce a footnote as follows: "Careful preparation of the zinc surface is necessary in order to assure satisfactory deposits. For suitable methods see the Tentative Recommended Practice for the Preparation of Zinc-Base Die Castings for Plating (ASTM Designation: B 252)."

Section 3.—Change the minimum thickness of copper required in the table to 0.0002 in. for Types F.Z., K.Z., and Q.Z.

Section 3(a).—Revise to read as follows: "Coatings on the finished articles shall conform to the following requirements as to thickness on significant surfaces."

Section 6.—In the last sentence add "visually" between "be" and "examined."

Section 7.—Revise to read as follows:

> The number of test specimens to be subjected to each test shall be agreed upon by the manufacturer and the purchaser. The thickness of chromium shall be determined in accordance with Section 8(*a*) and thickness of nickel and copper in accordance with Sections 8(*b*) or (*c*). The continuity of coating shall be determined in accordance with Section 9 after cut edges are coated with a suitable corrosion-resistant paint or lacquer.
>
> NOTE.—Wherever possible, thicknesses should be measured by magnetic methods on the maximum number of samples practicable since such measurements are non-destructive and inexpensive.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 2.

[2] 1952 Book of ASTM Standards, Part 2.

Recommended Practice for Preparation of High Carbon Steel for Electroplating (**B 242 – 51**):[2]

Section 5(f).—Change title of Section 5(*f*) to "Anodic Acid Treatment."

In line 2 of the first paragraph, change the word "etch" to "treatment."

In line 5 of the second paragraph, change the word "etching" to "treatment."

In line 17 of the second paragraph, delete the word "etch."

In lines 10 and 11 of the third paragraph, delete the word "etching."

In line 13 of the third paragraph, insert an asterisk after the word "mixture" and refer to U. S. Patent 2,334,699.

REVISION OF TENTATIVE AND ADOPTION AS STANDARD

The committee recommends the following revisions in the Tentative Recommended Practice for Preparation of and Electroplating on Aluminum Alloys (**B 253 – 51 T**)[2] and also recommends its adoption as standard as revised:

Section 4(b).—Add to this section the following:

Recently, a modification of the zincate solution has been developed for wrought and cast alloys, which in most applications gives more uniform and satisfactory results than the standard zinc immersion treatment. The modified zinc immersion procedure[8] has the following advantages:

(*1*) More uniform coverage by subsequent plating baths,

(*2*) Greater operating range for the "Double Immersion" surface conditioning treatment, and

(*3*) Improved resistance to corrosion on all plated aluminum alloys except for the 24S and 75S type alloys.

The modified solution is prepared by dissolving the zinc oxide in a sodium hydroxide solution in the usual way and cooling to room temperature. Before the bath is diluted to volume, a water solution of ferric chloride crystals and Rochelle salts is added. The bath should be stirred while the ferric chloride - Rochelle salts solution is added. The modified zincate solution should be made up as follows:

ZINC-IMMERSION SOLUTION	BATH I
Sodium hydroxide (commercial) (76 per cent Na_2O)	70 oz per gal (525 g per l)
Zinc oxide	13 oz per gal (100 g per l)
Ferric chloride crystals	0.13 oz per gal (1.0 g per l)
Rochelle salts	1.3 oz per gal (10 g per l)

This bath should also be operated under 80 F and for immersion times on the order of 30 sec to 1 min.

It is recommended that the modified zincate solution be utilized whenever the "double immersion" conditioning treatment is employed. Likewise, it will be found advantageous on all wrought and cast alloys, excepting of course the 24S and 75S types for corrosion-resistant applications.

Another variation of the modified zinc immersion procedure[9] has been developed for applications where the rinsing and drag-out are problems. This variation consists of reducing the bath viscosity by lowering the concentration of the principal components. At the same time, a low coating weight must be maintained by a closer control of operating conditions and by addition agents. Two typical dilute baths, which may be prepared in the usual manner, are as follows:

ZINC-IMMERSION SOLUTION	BATH II
Sodium hydroxide	6.7 oz per gal (50 g per l)
Zinc oxide	0.67 oz per gal (5 g per l)
Rochelle salts	6.7 oz per gal (50 g per l)
Ferric chloride crystals	0.27 oz per gal (2 g per l)
Sodium nitrate	0.13 oz per gal (1 g per l)

ZINC-IMMERSION SOLUTION	BATH III
Sodium hydroxide	16.0 oz per gal (120 g per l)
Zinc oxide	2.7 oz per gal (20 g per l)
Rochelle salts	6.7 oz per gal (50 g per l)
Ferric chloride crystals	0.27 oz per gal (2 g per l)
Sodium nitrate	0.13 oz per gal (1 g per l)

Bath III will provide a greater zinc reserve for high production work with only a small sacrifice in rinsing and dragout properties. When using these dilute solutions, the temperature must be maintained at 70 to 75 F and the immersion time must not exceed 30 sec.

Footnote 8.—Add the following footnote reference:

[8] The modified zinc immersion procedures described are the subjects of pending patent applications. When a U. S. patent or patents are issued, royalty free licenses will be granted thereunder.

Footnote 9.—Add the following footnote reference:

[9] The modified zinc immersion procedures described are the subjects of pending patent applications. When a U. S. patent or patents are issued, royalty free licenses will be granted thereunder.

Appendix, Section A2(d).—Change last item to read:

Container......steel, lined with a suitable plastic lining, such as Koroseal or carbon brick; preferably a combination of both.

Delete footnote 8.

Appendix, Section A2(e).—Change to read as follows:

(*e*) STANDARD ZINC-IMMERSION SOLUTION:

Sodium hydroxide	70 oz per gal (525 g per l)
Zinc oxide	13 oz per gal (100 g per l)
Time	½ to 1 min
Temperature	60 to 80 F (16 to 27 C)
Container	steel

MODIFIED ZINC IMMERSION SOLUTIONS:

I

Sodium hydroxide (commercial) (76 per cent Na_2O)	70 oz per gal (525 g per l)
Zinc oxide	13 oz per gal (100 g per l)
Ferric chloride crystals	0.13 oz per gal (1.0 g per l)
Rochelle salts	1.3 oz per gal (10 g per l)
Time	½ to 1 min
Temperature	60 to 80 F (16 to 27 C)
Container	steel

II

Sodium hydroxide	6.7 oz per gal (50 g per l)
Zinc oxide	0.67 oz per gal (5 per per l)
Rochelle salts	6.7 oz per gal (50 g per l)
Ferric chloride crystals	0.27 oz per gal (2 g per l)
Sodium nitrate	0.13 oz per gal (1 g per l)
Time	30 sec or less
Temperature	70 to 75 F (21 to 24 C)
Container	steel

III

Sodium hydroxide	16.0 oz per gal (120 g per l)
Zinc oxide	2.7 oz per gal (20 g per l)
Rochelle salts	6.7 oz per gal (50 g per l)
Ferric chloride crystals	0.27 oz per gal (2 g per l)
Sodium nitrate	0.13 oz per gal (1 g per l)
Time	30 sec or less
Temperature	70 to 75 F (21 to 24 C)
Container	steel

REVISION OF TENTATIVES

The committee recommends for revisions and continuation as tentative the following:

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 – 45 T**):[2]

Footnote 3.—Include in this footnote reference to the Recommended Practice for the Preparation of High-Carbon Steel for Electroplating (ASTM Designation: B 242).

Section 3.—Revise to read as follows: "Coatings on the finished articles shall conform to the following requirements as to thickness on significant surfaces."

Revise footnote b in Section 3 to read as follows: "The use of an extremely thin copper strike may cause operational difficulties such as corrosion of the basis metal during the transfer of parts."

Section 6.—In the last sentence add "visually" between "be" and "examined."

Section 7.—Revise to read as follows:

The number of test specimens to be subjected to each test shall be agreed upon by the manufacturer and purchaser. The thickness of chromium shall be determined in accordance with Section 8(*a*) and the thickness of nickel and copper in accordance with Sections 8(*b*) or (*c*). The continuity of coating shall be determined in accordance with Section 9 after cut edges are coated with a suitable corrosion-resistant paint or lacquer.

NOTE.—Wherever possible, thicknesses should be measured by magnetic methods on the maximum number of samples practicable since such measurements are non-destructive and inexpensive.

Section 8.—Revise Paragraph (*c*) to read as follows:

When agreed upon by the manufacturer and purchaser, the thickness of composite coatings of nickel and copper on steel with or without the chromium coating may be determined by the magnetic method. This method and its limitations are described in an Appendix to ASTM Methods A 219.

Section 12.—Add a footnote: "Nickel deposited by chemical reduction and otherwise meeting the requirements of the particular application may be accepted under this specification."

Tentative Specifications for Electrodeposited Coatings of Lead on Steel (**B 200 - 45 T**):[2]

Footnote 4.—Include in this footnote reference to the Recommended Practice for the Preparation of High Carbon Steel for Electroplating (ASTM Designation: B 242).

Section 6.—In the last sentence add "visually" between "be" and "examined".

Section 7.—Revise to read as follows:

The number of test specimens to be subjected to each test shall be agreed upon by the manufacturer and the purchaser. The thickness of lead and copper shall be determined in accordance with Section 8. The continuity of coating shall be determined in accordance with Section 9 after cut edges are coated with a suitable corrosion-resistant paint or lacquer.

NOTE.—Wherever possible, thicknesses shall be measured by magnetic methods on the maximum number of samples practicable since such measurements are non-destructive and inexpensive.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:[2]

Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 - 51 T),

Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 - 51 T),

Tentative Recommended Practice for Preparation of Zinc Base Die Castings for Electroplating (B 252 - 51 T), and

Tentative Recommended Practice for Preparation of and Electroplating on Stainless Steel (B 254 - 51 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following two tentatives, which have been published by the Society for two or more years, be continued without revision, because of test programs now in progress or revisions presently contemplated pertaining to recent information:

Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 51 T), and

Tentative Specifications for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die Cast Surfaces (B 201 - 49 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications, Papers, and Definitions (J. E. Stareck, chairman) has been active in reviewing specifications under its jurisdiction, resulting in actions recommended earlier in this report.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Performance Tests (W. L. Pinner, chairman) has continued outdoor exposure tests of electrodeposited copper-nickel-chromium coatings on high-carbon steel and lead coatings on steel. Exposure of a third set of panels in the copper-nickel-chromium program is planned in the near future. A new method of rating exposed panels has been devised. These are covered in a progress report of Subcommittee II which is appended.[4] A section has been formed under the chairmanship of J. E. Stareck to arrange for inspection of steel panels, cadmium and zinc plated, and subsequently given chromate surface treatments.

Subcommittee III on Conformance Tests (R. F. Ledford, chairman) has functioned part of the year under its new chairman; the work of all sections progressing.

A new section on "Adhesion Tests" has been authorized under the chairmanship of Fielding Ogburn, and this activity has thus been removed from the scope of an existing section.

Subcommittee IV on Electroplating Practice (Max Frager, chairman).—All existing sections have been working on their assignments through the year.

A subgroup under the chairmanship of Sam Spring has been appointed to survey the field of alkaline cleaning of metals with regard to a proposed paper on that subject. This work will be coordinated with the activities of Committee D-12 on Soaps and Other Detergents.

A task force headed by W. H. Safranek has been appointed to review the Standard Recommended Practice for the Preparation of High-Carbon Steel for Electroplating (B 242 – 51), and make recommendations for changes.

Section F on the Preparation of Plastics and Other Non-Conductors for Plating has been disbanded.

Subcommittee V on Supplementary Treatments for Electrodeposited Metallic Coatings (M. Darrin, chairman).—Several sections of Subcommittee V have been active during the year. A new section under the chairmanship of E. Roehl has been appointed and charged with surveying the field of surface treatments for lead.

Interest of Subcommittee V in chemical surface treatments for light metals, principally aluminum, has been evidenced.

This report has been submitted to letter ballot of the committee which consists of 112 voting members; 80 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. H SAMPLE,
Chairman

R. B. SALTONSTALL,
Secretary.

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[4] See p. 256.

REPORT OF SUBCOMMITTEE II ON PERFORMANCE TESTS

This report of Subcommittee II has been divided into three parts as follows: (1) copper-nickel-chromium deposits on high-carbon steel, (2) lead coatings on steel, and (3) methods of rating.

ATMOSPHERIC EXPOSURE OF COPPER-NICKEL-CHROMIUM DEPOSITS ON HIGH-CARBON STEEL

The most recent report of Subcommittee II was published in 1951.[1] That report furnished inspection data on the original set of copper-nickel-chromium plated panels which had been on exposure at various locations for approximately 3.5 yr. Reference was made at that time to a supplemental set of panels on which exposure tests had just been started. The present report includes virtually the final inspection data on the original set of panels and also includes inspection data for the supplementary set, after exposure times of 0.4, 1.4, and 2.4 yr.

Original Set of Cu-Ni-Cr Panels:

Table I presents the inspection data on these panels after 5.2 yr of exposure. The test is being continued at State College, Pa., where the effect of the rural atmospheres is so mild that a majority of the panels are still in good condition. By agreement with the inspection group, authority has been given to discontinue exposure tests on these panels at New York, N. Y. and Kure Beach, inasmuch as virtually all of them have approached a state of practically complete failure. At these two locations only, tests on these panels coated with 3 mils of total plate are being continued.

The two years of additional exposure on this set of panels at New York and Kure Beach has led to no necessity for modifying, within the scope of variables represented, the conclusions reached in the 1951 report of the Subcommittee. It is necessary, however, to emphasize that this statement refers to the original set only. Results from the supplemental set would lead to revisions in the conclusions. The type of atmosphere produced a profound effect, illustrated by comparing performance of panels at Pittsburgh, where virtually all panels had failed two years ago, with performance at State College, where up to the present time only the relatively thinner coatings have started to show corrosion effects.

At State College, panels coated with 1.0 and 1.5 mils of total plate thickness are starting to show corrosion, and the same trends are shown as were previously reported for the duplicated panels in the other locations. Panels containing a total plate thickness in excess of 1.5 mils are still in perfect, or nearly perfect, condition in the rural atmosphere at State College. In the 1951 report it was mentioned that the application of a nickel coating in two steps with the first layer buffed, or annealed and buffed, was highly beneficial in a seacoast environment, but was of no benefit in industrial atmospheres. It is seen, by comparing lot numbers 4NA and 4B with the other lots in which the total thickness is 1.0 mils, that the double nickel

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 204 (1951).

TABLE I.—INSPECTION DATA ORIGINAL SET Cu-Ni-Cr.

NOTE.—(b) = buffed; (ba) = annealed and buffed; (WA) = wetting agent; (pH) = high pH bath; SB = semi-bright; B = bright. [a] Incorrectly listed as SAE 9112 in 1951.

Lot	Steel (SAE)	Coating, mils			5.2 yr	
		Cu	Ni	Total	Rating	Description
KURE BEACH, N. C.						
No. 1	1095	...	1.0	1.0	0	xRs; xcR; xR
No. 2		0.1	0.9	1.0	0	xRs; xcR; xB
No. 3		0.5	0.5	1.0	0	xRs; xcR; xB
No. 4		0.5 (b)	0.5	1.0	0	xRs; xcR; xR
4NA		...	0.5 (b) +0.5	1.0	8	sRs; scR; sSp
4B		...	0.5 (ba) +0.5	1.0	8	xRs; scR; sSp
No. 5X		...	1.0 (pH)	1.0	0	xRs; xcR; xB
No. 6X		0.1	0.9 (pH)	1.0	0	xRs; xcR; xB
No. 5	1095	...	1.5	1.5	5	iRs; icR; ipR
No. 6		0.1	1.4	1.5	4	iRs; icR; xcR
No. 7		0.5	1.0	1.5	0	xRs; xcR; xB
No. 8		1.0	0.5 (WA)	1.5	0	xRs; xcR; xB
No. 9		1.0 (b)	0.5	1.5	0	xRs; xcR; xB
No. 9NA	1095	...	−1.0 (b) +0.5	1.5	9	sRs; vscR; sSp
No. 10		...	2.0	2.0	8	sRsvscR; sSp
No. 11		0.1	1.9	2.0	6	iRs; ipR; icR
No. 12		0.5	1.5	2.0	5	iRs; ipR; icR
No. 13		1.0	1.0	2.0	2	xRs; xpR; xcR
No. 14		1.0 (b)	1.0	2.0	1	xRs; xpR; xcR
No. 14NA		...	1.0 (b) +1.0	2.0	9	sRs; spR; scR
No. 14B		...	1.0 (ba) +1.0	2.0	9	sRs; spR; vscR
No. 15		1.5	0.5	2.0	0	xRs; xpR; xcR
No. 16		1.5 (b)	0.5	2.0	1	xRs; xpR; xcR
No. 10X	1095	...	3.0	3.0	9	vsRs; vspR; vscR
No. 14X		1.0	2.0	3.0	8	sRs; vspR; vscR
No. 16X		1.5	1.5	3.0	8	sRs; vspR; vscR
No. 25	4130	...	1.5	1.5	3	xRs; xpR; xcR
No. 26		0.1	1.4	1.5	7	vsRs; vspR; scR; sB
No. 27		0.5	1.0	1.5	0	vxcR
No. 28	1095	...	2.0 SB	2.0	0	xpR; scR; xB
No. 29		1.0	1.0 SB	2.0	2	xRs; xpR; xcR
No. 29 NA		...	1.0 (b) +1.0 SB	2.0	2	xRs; xpR; xcR
No. 30	1095	1.0	1.0 B	2.0	1 3	xRs; xpR; xcR xRs; xpR; xcR (2 panels)
No. 31		1.0 (b)	1.0 B	2.0	9	vspR (2 panels)
No. 32		0.1	0.9 +1.0 B	2.0	6	sRs; spR; vscr; sB
No. 33		0.1	0.9 (b) +1.0 B	2.0	8	xSp; vsB
No. 34	1095 high polish	0.1	0.9 (b) +1.0 B	2.0	9	xSp
NEW YORK, N. Y.						
No. 1	1095	...	1.0	1.0	2	sRs; ipR; xSp; seZ; iR
No. 2		0.1	0.9	1.0	1	xRs; xpR; xcR; xSp; seZ
No. 3		0.5	0.5	1.0	0	xpR; xcR; xRs
No. 4		0.5 (b)	0.5	1.0	0	xpR; xcR; xRs
No. 4NA		...	0.5 (b) +0.5	1.0	2	xpR; sRs; xSp; xcR; xZ; xW
No. 4B		...	0.5 (ba) +0.5	1.0	2	xpR; sRs; xSp; xcR; xB; xW
No. 5X		...	1.0 (pH)	1.0	2	xpR; sRs; xSp; xcR; xZ; xW
No. 6X		0.1	0.9 (pH)	1.0	1	xpR; sRs; xSp; xcR; xZ; xW
No. 5		...	1.5	1.5	2	xpR; scR; sRs; xSp; seZ
No. 6	1095	0.1	1.4	1.5	2	xpR; scR; sRs; xSp; seZ
No. 7		0.5	1.0	1.5	0	xpR; xcR; xRs; xSp
No. 8		1.0	0.5 (WA)	1.5	0	xpR; xcR; xRs; xSp
No. 9		1.0 (b)	0.5	1.5	0	xpR; xcR; xRs; xSp

TABLE I.—INSPECTION DATA ORIGINAL SET Cu-Ni-Cr—*Continued.*

Lot	Steel (SAE)	Coating, mils			5.2 yr	
		c	Ni	Total	Rating	Description
No. 9NA	1095	...	−1.0 (b) +0.5	1.5	2	xpR; scR; sRs; xSp; seZ
No. 10		2.0	2.0	2.0	2	xpR; scR; sRs; xSp; seZ
No. 11		0.1	1.9	2.0	2	xpR; scR; sRs; xSp; seZ
No. 12		0.5	1.5	2.0	0	xpR; xcR; xRs; xSp
No. 13		1.0	1.0	2.0	0	xpR; xcR; xRs; xSp
No. 14		1.0 (b)	1.0	2.0	0	xpR; xcR; xRs; xSp
No. 14NA		...	1.0 (b) +1.0	2.0	2	xpR; icR; sRs; xSp
No. 14B			1.0 (ba) +1.0	2.0	2	xpR; icR; sRs; xSp; seZ
No. 15		1.5	0.5	2.0	0	xpR; xcR; xRs; xSp
No. 16		1.5 (b)	0.5	2.0	0	xpR; xcR; xRs; xSp
No. 10X	1095	...	3.0	3.0	2	sRs; ipR; xSp
No. 14X		1.0	2.0	3.0	2	sRs; ipR; xSp
No. 16X		1.5	1.5	3.0	1	sRs; ipR; xSp
No. 25	4130	...	1.5	1.5	1	iRs; ipR; xSp; seZ
No. 26		0.1	1.4	1.5	2	iRs; xpR; icR; xSp; seZ
No. 27		0.5	1.0	1.5	0	xpR; xcR; xRs; xSp
No. 28	1095	...	2.0 SB	2.0	1	spR; icR; iRs; (peeled)
No. 29		1.0	1.0 SB	2.0	0	xpR; xcR; xRs; xSp
No. 29NA		...	1.0 (b) +1.0 SB	2.0	2	sRs; ipR; xSp; seZ
No. 30	1095	1.0	1.0 B	2.0	0	xRs; xcR; iSp
No. 31		1.0 (b)	1.0 B	2.0	0	xRs; xcR; iSp
No. 32		0.1	0.9 +1.0 B	2.0	1	sRs; icR; xSp; iW
No. 33		0.1	0.9 (b) +1.0 B	2.0	1	sRs; IcR; xSp; iW
No. 34	1095 high-polish	0.1	0.9 (b) +1.0 B	2.0	1	sRs; icR; xSp; iW
STATE COLLEGE, PA.						
No. 1	1095	...	1.0	1.0	5	ipR; scR
No. 2		0.1	0.9	1.0	4	spR; icR; sB
No. 3		0.5	0.5	1.0	0	sRs; xcR; xpR; isB
No. 4		0.5 (b)	0.5	1.0	1	sRs; xcR; xpR; isB
No. 4NA		...	0.5 (b) +0.5	1.0	7	sSp; xcR
No. 4B		...	0.5 (ba) +0.5	1.0	10	iSp
No. 5X		...	1.0 (pH)	1.0	6	iSp; spR; vsRs
No. 6X		0.1	0.9 (pH)	1.0	3	vsSp; iRs; xB
No. 5	1095	...	1.5	1.5	9	xSp; spR
No. 6		0.1	1.4	1.5	10	iSp
No. 7		0.5	1.0	1.5	6	iSp; icR; vsB; sB
No. 8		1.0	0.5 (WA)	1.5	3	iSp; icr; iRs; xB
No. 9		1.0 (b)	0.5	1.5	4	iSp
No. 9NA	1095	...	−1.0 (b) +0.5	1.5	10	iSp
No. 10		...	2.0	2.0	10	iSp
No. 11		0.1	1.9	2.0	10	iSp
No. 12		0.5	1.5	2.0	10	iSp
No. 13		1.0	1.0	2.0	10	iSp
No. 14		1.0 (b)	1.0	2.0	10	iSp
No. 14NA		...	1.0 (b) +1.0	2.0	10	iSp
No. 14B		...	1.0 (ba) +1.0	2.0	10	iSp
No. 15		1.5	0.5	2.0	8	iSp; fsB; vscr
No. 16		1.5 (b)	0.5	2.0	9	iSp; fsb
No. 10X	1095	...	3.0	3.0	10	iSp
No. 14X		1.0	2.0	3.0	10	iSp
No. 16X		1.5	1.5	3.0	10	iSp
No. 26	950	...	1.5	1.5	8	iSp; spR
No. 26		0.1	1.4	1.5	10	iSp
No. 27		0.5	1.0	1.5	9	iSp; eR

TABLE I.—INSPECTION DATA ORIGINAL SET Cu-Ni-Cr—*Concluded.*

Lot	Steel (SAE)	Coating, mils			5.2 yr	
		Cu	Ni	Total	Rating	Description
No. 28	1095	...	2.0 SB	2.0	10(2) 2(2)	iSp; xF; eR
No. 29		1.0	1.0 SB	2.0	9	iSp; isB
No. 29NA		...	1.0 (b) +1.0 SB	2.0	10	iSp
No. 30	10)	1.0	1.0 B	2.0	8	iSp; xsB
No. 31		1.0 (b)	1.0 B	2.0	6	iSp
No 32		0.1	0.9 +1.0 B	2.0	6	xSp; spR
No. 33		0.1	0.9 (b) +1.0 B	2.0	6	xSp; spR
No. 34	high-polish 1095	0 1	0.9 (b) +1.0 B	2.0	10	iSp

TABLE II.—INSPECTION DATA, PROGRAM NO. 2 Cu-Ni-Cr.

NOTE.—A = acid; C = cyanide; P = pyrophosphate; (b) = buffed; [a] = Nil Cu; [b] = 10 mg Cu per 1.; [c] = 25 mg Cu per 1. [d] Incorrectly listed as SAE 9112 in 1951.

NEW YORK, N. Y.

Lot	Steel (SAE)	Coating, mils			0.4 yr Rating	1.4 yr		2.4 yr.	
		Cu	Ni	Total		Rating	Description	Rating	Description
No. 1	950^d	...	1.0	1.0	7	2	xpR; iRs; xSp; xeZ	1	iRs; vscR; xSp; xpR; eZ; iW; ipR
No. 2		0.1 C	0.9	1.0	7	2	ipR; sRs; xSp; fiB; xeR	3	vsRs; scR; xSp; iB; eZ; iW
No. 3		...	1.5	1.5	8	4	ipR; sRs; xSp; xeZ; vseR	2	iRs; icR; xSp; vsB; eZ; iW
No. 4		0.1 C	1.4	1.5	9	4	xSp; seZ	7	sRs; scR; xSp; vseZ; iW; ipR
No. 5		0.1 C + 0.4 A	1.0	1.5	8	3	ipR; xSp; iS; xeR	5	iRs; scR; xSp; seZ; iW; ipR
No. 6		0.2 C + 0.3 A	1.0	1.5	9	3	ipR; xSp; iS; xeR	5	iRs; scR; xSp; ipR; seZ; iW
No. 7		0.2 C	1.0	1.2	8	4	ipR; xSp; iS; xeR	6	iRs; ipR; xSp; vseZ; iW
No. 8		0.5 C	1.0	1.5	8	4	ipR; xSp; iS; xeR	6	sRs; ipR; xSp; vseZ; iW
No. 9		0.05 C 0.5 P	1.0	1.5	9	4	ipR; xSp; iS; xeR	6	sRs; ipR; xSp; vseZ; iW
No. 10		0.2 C + 0.3 A (b)	1.0	1.5	9	3	ipR; xSp; iS; xeR	5	xRs; ipR; xSp; ieZ; iW
No. 11		0.05 C 0.5 P (b)	1.0	1.5	8	4	ipR; xSp; iS; xeR	7	xRs; iSp; spR
No. 1	1010	...	1.5^a	1.5	8	2	ipR; xSp; iS; xeR; vfxB	2	xRs; xSp; xpR; iB; xeZ; sW
No. 2		...	1.5^b	1.5	8	3	ipR; xSp; iS; xeR	3	sRs; iSp; ipR; ieZ; sW
No. 3		...	1.5^c	1.5	8	3	ipR; xSp; iS; xeR	5	vscR; xSp; iZ; sW

KURE BEACH, N. C.

Lot	Steel (SAE)	Coating, mils			0.4 yr Rating	1.4 yr		2.4 yr.	
		Cu	Ni	Total		Rating	Description	Rating	Description
No. 1	950	...	1.0	1.0	9	3	xpR; ieR; iRs; iB; xF	3	iRs; ipR; xB; xeF
No. 2		0.10	0.9	1.0	9	4	xpR, ieR; iRs; iB; iF	2	iRs; xpR; xB; xeF
No. 3		...	1.5	1.5	9	6	spR; sRs; iF	5	iRs; spR; sB, xeF
No. 4		0.1 C	1.4	1.5	9	7	spR; sRs; sF	5	iRs; spR; scR; sB; xeF
No. 5		0.1 C + 0.4 A	1.0	1.5	9	5	spR; seR; sRs; sB; sF	4	iRs; spR; icR; iB; xeF
No. 6		0.2 C + 0.3 A	1.0	1.5	9	5	spR; scR; sRs; sB; sF	2	xRs; xcR; xB; xeF
No. 7		0.2 C	1.0	1.2	9	4	ipR; ieR; iRs; iB; sF	1	xRs; xcR; xB; xeF
No. 8		0.5 C	1.0	1.5	9	8	spR; sRs; sB; scR	8	sRs; spR; vsB; xeF
No. 9		0.5 P	1.0	1.5	9	6	ipR; sRs	5	iRs; ipR; vsB
No 10		0.2 C + 0.3 A(b)	1.0	1.5	9	6	spR; seR; sRs; sF	4	iRs; ipR; icR; iB; xeF
No. 11		0.5 P (b)	1.0	1.5	9	6	spR; seR; sRs; sF	6	iRs; ipR; vscR; sB
No. 1	1010	...	1.5^a	1.5	9	5	ipR; seR; sRs; sB; sF	3	iRs; ipR; icR; sB; xeF
No. 2		...	1.5^b	1.5	8	5	ipR; seR; sRs; sB	3	iRs; ipR; icR; sB; xeF
No. 3		...	1.5^c	1.5	8	3	ipR; ieR; iRs; iB	2	xRs; xpR; icR; iB; xeF

coating found to be so beneficial in a seacoast environment is equally beneficial in the rural exposure at State College. Further in this regard, lot 4B indicates that if the underlying nickel is annealed and buffed and then followed by a second nickel plate, this type of coating is exceptionally good in a rural atmosphere, as this set of panels with a total thickness of 1.0 mils shows no corrosion of the base metal at all after 5 yr and exhibits only a mild condition of surface pitting of the plated coating.

A comparison of the ratings published in this report with those published in 1951 reveals somewhat higher values at this time in a number of instances. It is possible that several heavy rainfalls, just preceding the inspection, may have contributed somewhat to the altered values. Another consideration is the fact that the inspection group de-emphasized surface pitting with respect to the rating numbers. This is discussed in somewhat more detail later in the report.

The system of abbreviations used to supplement the rating number record and to define the types of deterioration is given below:

TYPES OF FAILURE

R = Corrosion (rusting) of the basis metal. (Permanent or massive type of basis metal corrosion such as that in pinholes, bare or flaked areas, or in craters of broken blisters.)
Rs = Stain due to basis metal corrosion products, such as rust stain, which can be removed readily with a damp cloth or chamois and mild abrasive revealing a sound bright surface.
S = Stains or spots other than that of *obvious* basis metal corrosion products.
Sp = Surface pits. Corrosion pits probably not extending through to the basis metal—that is, absence of *obvious* basis metal corrosion products bleeding therefrom.
F = Flaking or peeling of deposit.
B = Blistering.
C = Cracking.
Z = Crazing.
W = Crow's feet.

DEGREE OR EXTENT OF PINHOLE RUSTING, STAINING, SURFACE PITTING, FLAKING, ETC.

vs = very slight amount.
s = slight amount.
i = intermediate or moderate amount.
x = excessive amount.

DESCRIPTION OF BLISTERS

s = less than about 0.5 mm in diameter.
i = about 0.5 to 2.0 mm in diameter.
x = greater than about 2.0 mm in diameter.

vf = 5 or fewer.
f = 5^+ to 25.
i = 10^+ to 25.
m = 25^+ to 50.
vm = over 50.

DESCRIPTION OF LOCATION OF DEFECTS

e = edge
g = general

EXAMPLES

(1) ipR, vscR, sRs = a moderate amount of pin-point type rusting, a very slight amount of crater-type rusting, and a slight amount of rust stain which can be readily removed.

(2) iSp, iS, iiW, seZ = moderate surface pitting, moderate staining (other than rust), an intermediate number of moderate size crow's feet, a small amount of edge crazing.

(3) xcR, xRs = excessive crater-type rust spots and an excessive amount of removable rust over a sound, bright surface.

Supplemental Cu-Ni-Cr Exposure Tests:

In 1951[1] this set of panels was referred to as the supplemental program. It will hereafter be referred to as Performance Test Program No. 2.

Test Program No. 2 was undertaken with the purpose of spot checking some of the results of the original program obtained from those combinations of coatings which seemed to be in conflict with data available from other sources.[2] Some question was raised as to the possible effect of the thickness of the cyanide copper strike used under the conventional acid copper plate in the original set of panels. It was also considered that there was merit in investigating copper deposited from alkaline as well as acid baths. A third consideration included was the effect of copper as an impurity in the nickel baths on the corrosion resistance of nickel deposits.

The types of steel and the various combinations and thicknesses of plated coatings, together with the ratings and description of the types of corrosion, are summarized in the inspection data presented in Table II.

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[2] Lux and Blum, *Journal of Research*, Nat. Bureau Standards, Vol. 34, p. 295 (1945); Blum and Strausser, *Journal of Research*, Nat. Bureau Standards, Vol. 24, p. 443 (1940); Blum, Strausser, and Brenner, *Journal of Research*, Nat. Bureau of Standards, Vol. 13, p. 331 (1934) Thomas and Blum, *Transactions*, Am. Electrochemical Soc., Vol. 48, p. 69 (1925), Vol. 52, p. 271 (1927); Jacquet, *Bulletin*, Soc. Française Electriciens, Vol. 2, p. 631 (1932); Kraus, *Korrosion und Metallschutz*, Vol. 4, p. 153 (1928); Wirshing, *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 8, pp. 9–12, 26–31 (1932).

Prior to plating, the steel had been polished with a grit sequence as follows: 100, 120, 150 dry, 150 grease. Profilometer data were taken from polished steel panels as a check on the uniformity of the operation. The panels were prepared at the Research Laboratory of the General Motors Corporation in their small scale plating line with the exception of lot Nos. 9 and 11 on which pyrophosphate copper was plated at the laboratory of United Chromium, Inc. Control of thickness was effected by standardizing current density-time-temperature relationship for each individual plating bath by means of preliminary control panels. These control panels were mounted on racks and subsequently used in identical manner for processing the test panels. For each plating bath and each thickness required, the operating conditions were established and checked by means of thickness measurements. These thickness measurements were made by a composite thickness magnagage designed and calibrated by National Bureau of Standards to measure accurately copper-nickel composite coatings to a total thickness of 2.75 mils. The pre-establishing of operating conditions was necessary because the plating procedure does not permit intermediate plate thickness determinations. All buffing of the panels and the nickel and chromium plating of the panels was done at General Motors Corp. Research Laboratory. These panels were subsequently exposed at New York and Kure Beach in early February 1950.

TABLE III.—COMPARISON OF DUPLICATION OF ORIGINAL AND PROGRAM 2 LOTS AFTER 2.5 YR.

New York				Kure Beach			
Lot		Rating		Lot		Rating	
Original	Program 2	Original	Program 2	Original	Program 2	Original	Program 2
No. 1.	No. 1	1	1	No. 1	No. 1	3	3
No. 2.	No. 2	2	3	No. 2	No. 2	2	2
No. 25.	No. 3	3	2	No. 25	No. 3	5	5
No. 26.	No. 4	3	7	No. 26	No. 4	6	5

TABLE IV.—TEST PANELS FOR PERFORMANCE TEST PROGRAM NO. 3.

Note: All panels to be chromium plated to a thickness of 0.01 mils.

Lot	Steel (SAE)	Coating, mils			
		Cyanide Copper	Thiourea Acid Copper	Bright Nickel	Total
No. 1.....	950	...	...	1.5	1.5
No. 2.....		0.05	0.95	0.5	1.5
No. 3.....		0.2	0.8	0.5	1.5
No. 4.....		0.05	0.45	1.0	1.5
No. 5.....		0.2	0.3	1.0	1.5
			Acid Copper	Watt's Nickel	
No. 6.....	950	0.05	0.45	1.0	1.5
No. 7.....	950	0.1	...	1.4	1.5

The following are the details of procedure used in plating the Test Program No. 2 set of panels:

SOLUTIONS:

Cleaner G. M. 2401-M

Soda ash	35 per cent
Trisodium phosphate	55 per cent
Sodium hydroxide	10 per cent
	6 oz per gal
Current density	60 amp per sq ft
Temperature	160–200 F

Cyanide Copper (also used as strike before acid and pyrophosphate copper)

Copper as metal	2.26 oz per gal
Sodium hydroxide	0.95 oz per gal
Carbonates	3.92 oz per gal
Free cyanide	0.53 oz per gal
Current density	15 amp per sq ft
Temperature	120–125 F.

Acid Copper Solution

Copper sulfate	32 oz per gal
Sulfuric acid	4 oz per gal
Current density	30 amp per sq ft
Temperature	Room

Pyrophosphate Copper Solution

Copper as metal	4.2 oz per gal
Pyrophosphate	30.0 oz per gal
Ammonia	0.12 oz per gal
pH	8.4
Current density	30 amp per sq ft
Temperature	140 F

Watts Nickel Solution

Nickel sulfate	40 oz per gal
Nickel chloride	5 oz per gal
Boric acid	5 oz per gal
Hydrogen Peroxide as required to control pitting	
pH	2.5
Current density	30 amp per sq ft
Temperature	120–125 F

Chromium Plating Solution

Chromic acid	32 oz per gal
Chromate/sulfate ratio	100/1
Current density	150 amp per sq ft
Temperature	110 F

Panels were mounted back to back, four to a rack with a frame around the outside.

CLEANING AND PLATING CYCLES:

Step	Nickel-Chromium on Steel[a]	Copper-Nickel-Chromium on Steel[b]
No. 1	Cathodic clean	Cathodic clean
No. 2	Hand scrub	Hand scrub
No. 3	Anodic clean	Anodic clean
No. 4	Cold rinse	Cold rinse
No. 5	1:1 HCl dip	1:1 HCl dip
No. 6	Cold rinse	Cold rinse
No. 7	Nickel plate	Copper cyanide plate
No. 8	Buff	Cold rinse
No. 9	Cathodic clean	1:1 HCl dip
No. 10	Hand scrub	Cold rinse
No. 11	Cold rinse	Acid copper plate or pyrophosphate copper plate
No. 12	1:1 HCl dip	Buff
No. 13	Cold rinse	Cathodic clean
No. 14	Chromium plate	Hand scrub
No. 15		Cold rinse
No. 16		1:1 HCl dip
No. 17		Cold rinse
No. 18		Nickel plate
No. 19		Buff
No. 20		Cathodic clean
No. 21		Hand scrub
No. 22		Cold rinse
No. 23		1:1 HCl dip
No. 24		Cold rinse
No. 25		Chromium plate

[a] Used for lot Nos. 1, 3, of 950 steel and 1, 2, 3 of 1010 steel.

[b] Used for lot Nos. 9, 10, 11. Omit steps 12 through 14 for lot Nos. 5, 6, and 9. Omit step Nos. 11 through 17 for lot Nos. 2, 4, 7, 8.

It is interesting first to compare the ratings of the No. 2 program with those of the original set on lots which duplicate each other, after both had been on exposure for approximately 2.5 yr. These data are presented in Table III and, with the exception of one lot at New York, there is a rather faithful duplication of results. In the Performance Test Program No. 2, three sets of panels, made of SAE 1010 steel, were plated with nickel from three solutions, two of which contained copper as an impurity. It would appear in the data that copper in amounts up to 25 mg per l apparently has a negligible effect.

It is evident from an inspection of the data on the No. 2 lot of panels listed in Table II that the results, as far as the effect of copper is concerned, are not in agreement with those of the original set. While results are confirmatory on the whole, there is definite disagreement on the matter of the value of nickel-chromium *versus* copper-nickel-chromium deposits. These results received intensive study by Section A, which subsequently reported to Subcommittee II in part as follows:

> "Comparison of ratings between the original exposure group and the No. 2 set is incongruous. It is recommended by this section that additional test panels be prepared for performance testing to further evaluate copper-nickel-chromium deposits on a broader scope, with special emphasis on control and application of copper undercoats. It is further recommended by this section that a regular washing cycle be incorporated in performance test outdoor exposure procedures on portions of exposed panels to more nearly simulate service conditions."

Subcommittee II, and subsequently Committee B-8, approved this recommendation so that at the time of writing this report additional panels are being prepared and will be placed on exposure in the spring of 1953. This additional set will consist of panels on which plate thicknesses will be applied as specified in the data presented in Table IV. The steel to be used will be flat polished to a 5 rms finish. In lot Nos. 2 to 5 inclusive the copper will be buffed. In lot Nos. 6 and 7, designed

TABLE V.—SUMMARY OF INSPECTION DATA ON THE ATMOSPHERIC EXPOSURE TESTS OF ELECTROPLATED LEAD COATINGS.

Note.—P—Number of pinholes up to 25; MP—pinholes, more than 25; S—percentage of area stained, coating intact; R—percentage of flaky rust of base metal; Sl—slight; (d) = dark; N.D. = no data. Thickness of copper plate in all cases, 0.000015 in.

NEW YORK, N. Y.

	Nominal Coating Thickness, mils	Number of Panels Averaged	Upper Surface				Under Surface	
			5.4 yr Average (1949)	7.45 yr Average (1951)	5.4 yr Range	7.45 yr Range	5.4 yr Average	7.45 yr Average
Sulfamate Pb on steel	0.50	4	MP-8S	MP-20S	0-20S	5-50S	MP-15S	MP-20S
	1.00	4	...	...	0	0	...	...
Fluoborate Pb on steel	0.08	2	100R	100R	0	0	MP-100S-3R	MP-100S-7R
	0.25	4	MP-100S-2R	MP-100S-60R	0- 10R	10-90R	MP- 60S	MP- 70S
	0.50	4	MP- 90S	MP- 90S- 5R	80- 100S	2- 10R	MP- 20S	MP- 40S
	0.75	4	MP- 55S	MP- 55S- 1R	15- 80S	20- 85S	MP- 40S	MP- 40S
	1.00	4	MP- 2S	MP- 1S	0- 5S	0- 2S	MP- 10S	MP- 15S
	2.00	8	...	...	0	0	...	...
Sulfamate Pb on Cu-plated steel	0.50	4	...	...	0	0	MP-6S	MP-5S
	1.00	4	...	...	0	0	...	...
Fluoborate Pb on Cu-plated steel	0.08	2	−100R	100R	0	0	MP-100S-25R	100R
	0.25	4	MP-25S	MP-100S-10R	10-50S	1-20R	MP- 5S	MP-4S
	0.50	4	...	MP- 1S	0	0- 3S	...	MP
	0.75	4	...	...	0	0	...	...
	1.00	4	...	...	0	0	...	...
	2.00	8	...	...	0	0	...	...
Sulfamate Pb on sheet copper	0.25	3	...	...	0	0	...	...
	0.50	3	...	...	0	0	...	...
Fluoborate Pb on sheet copper	0.25	3	...	...	0	0	...	...
	0.50	3	...	...	0	0	...	...
Sulfamate Pb-Sn (2 per cent Sn) on steel	1.00	3	MP-8S	MP-8S	5-15S	2-20S	MP-1S	MP-3S
Sulfamate Pb-Sn (10 per cent Sn) on steel	1.00	3	MP-5S	MP-5S	0-10S	0-15S	...	...
Commercial hot-dipped terne (20 per cent Sn)	0.17	3	MP-100S	−100R	0	50-100R	MP-35S	MP-100S-5R
Commercial hot-dipped lead (2.5 per cent Sn, 2 per cent Sb)	0.40	3	MP-90S	MP-95S-70R	80-95S	50-100R	MP-20S	MP-20S
Electroplated zinc on steel	0.20	3	100R	100R	0	0	100R	100R
Hot dipped zinc on steel	0.75	3	100R	100R	0	0	MP-100S-35R	100R
Bare steel	...	4	100R	100R	0	0	100R	100R
Fluborate Pb on steel (damaged)	0.50	3	Spread	Bad Spread			MP-5S	MP-1S
	1.0	3	Spread	Spread			...	...
Fluoborate Pb on Cu-plated steel (damaged)	0.50	3	Sl. Spread	Bad Spread			...	MP-2S
	1.00	3	Sl. Spread	Spread			...	...

STATE COLLEGE, PA.

	Nominal Coating Thickness, mils	Number of Panels Averaged	Upper Surface					Under Surface		
			5.2 yr Average (1949)	6.15 yr Average (1950)	7.25 yr Average (1951)	5.2 yr Range	7.25 yr range	5.2 yr Average	6.15 yr Average	7.25 yr Average
Sulfamate Pb on steel	0.50	4	100R	100R	100R	0	100R	MP-<1S	MP-8S	MP-7S-<1R
	1.0	4	MP-2S	MP-30S-2R	MP-95S-7R	1-3S	90-100S, 2-15R	...	...	...
Fluoborate Pb on steel	0.08	2	100R	100R	100R	0	0	100R	100R	100R
	0.25	4	100R	100R	100R	0	0	100R	100R	100R
	0.50	5	100R	100R	100R	0	0	100R	MP-35S-6R	MP-75S- 9R
	0.75	4	MP-95S-70R	MP-100S-85R	100R	90-100S, 40-90R	0	10P	MP-15S	MP- 8S-<1R
	1.00	4	MP-20S- 1R	MP- 65S- 6R	MP-48S-50R	3- 40S, 0- 2R	95-100S, 30-60R	...	1P	1P
	2.00	8	...	...	1P	0	0-5P	...	...	...
Sulfamate Pb on Cu-plated steel	0.50	4	MP-35S-3R	MP-90S-25R	MP-100S-95R	20-40S, 2-3R	90-95R	MP-1S	MP-4S	MP-6S-1R
	1.00	4	MP-	MP-	MP-	0	0	...	2P	2P
Fluoborate Pb on Cu-plated steel	0.08	2	100R	100R	100R	0	0	100R	100R	100R
	0.25	4	100R	100R	100R	0	0	MP-70S-30R	MP-100S-30R	MP-100S-60R
	0.50	4	MP-30S-5R	MP-95S-55R	100R	10-40S, 2-8R	95-100R	MP- 5S- 1R	MP- 20S- 2R	MP- 40S- 3R
	0.75	4	MP-<1S	MP- 2S	MP-25S-5R	0- 1S	20-30S, 3-5R	4P	8P	MP-
	1.00	4	MP-	MP-	MP- 1S	0- 1S	0-3S	1P	20P	12P
	2.00	8	...	...	...	0	0	...	...	...
Sulfamate Pb on sheet copper	0.25	3	20S(d)	15S(d)	30S(d)	10-20S	20-40S	...	...	...
	0.50	3	...	1S(d)	...	0	0	...	...	...
Fluoborate Pb on sheet copper	0.25	3	15S(d)	7S(d)	25S(d)	5-20S	20-30S	...	...	...
	0.50	3	...	...	...	0	0	...	...	...
Sulfamate Pb-Sn (2 per cent Sn) on steel	1.00	3	100R	100R	100R	0	0	MP-70S-15R	MP-100S-12R	MP-100S-15R
Sulfamate Pb-Sn (10 per cent Sn) on steel	1.00	3	MP-100S-70R	MP-100S-70R	MP-100S-80R	15-100R	40-100R	...	...	...
Commercial hot-dipped terne (20 per cent Sn)	0.17	3	100R	100R	100R	0	0	100R	100R	100R
Commercial hot-dipped lead (2.5 per cent Sn, 2 per cent Sb)	0.40	3	MP-100S-50R	MP-100S-50R	MP-100S-70R	30-80R	50-100R	MP-90S-50R	MP-100S-80R	MP-100S-95R
Electroplated zinc on steel	0.20	3	100R	100R	100R	0	0	2R	100R	30R
Hot-dipped zinc on steel	0.75	3	<1R	2R	MP-100S-3R	0	0	...	...	...
Bare steel	...	4	100R	100R	100R	0	0	100R	100R	100R
Fluoborate Pb on steel (damaged)	0.50	3	100R	100R	100R			MP-30S-5R	MP-70S-10R	MP-95S-20R
	1.0	3	Spread	Spread	Spread			...	3P	MP-
Fluoborate Pb on Cu-plated steel (damaged)	0.50	3	Bad Spread	Bad Spread	100R			MP-2S-<1R	MP-5S-1R	MP-20S-1R
	1.00	3	Very Sl. Spread	Sl. Spread	Spread			...	2P	...

KURE BEACH, N. C.

	Nominal Coating Thickness, mils	Number of Panels Averaged	Upper Surface					Under Surface		
			5.38 yr Average (1949)	6.4 yr Average (1950)	7.4 yr Average (1951)	5.38 yr Range	7.4 yr Range	5.38 yr Average	6.4 yr Average	7.4 yr Average
Sulfamate Pb on steel	0.50	4	100R	100R	100R	0	0	100R	100R	100R
	1.00	4	MP-78S-28R	MP-95S-35R	MP-95S-85R	70-90S, 20-30R	80-90R	MP-10S-<1R	MP-25S-3R	MP-30S-<1R
Fluoborate Pb on steel	0.08	2	100R	100R	100R	0	0	100R	100R	100R
	0.25	4	100R	100R	100R	0	0	100R	100R	100R
	0.50	4	100R	100R	100R	0	0	100R	100R	100R
	0.75	4	100R	95R	100R	0	0	100R	40R	100R
	1.00	4	98R	60R	100R	90-100R	0	90R	MP-98S-4R	90R
	2.00	8	MP-3S-<1R	MP-6S-<1R	MP-8S-3R	2-10S, 0-2R	5-25S, 0-15R	MP-5S-<1R	MP-6S	MP-12S-<1R
Sulfamate Pb on Cu-plated steel	0.50	4	100R	100R	100R	0	0	MP-90S	MP-100S-40R	93R
	1.00	4	MP-40S-4R	MP-50S-8R	MP-60S-20R	10-80S, 0-10R	15-95S, 2-50R	MP-25S-1R	MP-18S	MP-14S-2R
Fluoborate Pb on Cu-plated steel	0.08	2	100R	100R	100R	0	0	100R	100R	100R
	0.25	4	100R	100R	100R	0	0	100R	100R	100R
	0.50	4	100R	100R	100R	0	0	MP-70S-30R	MP-98S-40R	MP-90S-75R
	0.75	4	MP-90S-80R	MP-90S-70R	92R	60-100R	80-100R	MP-11S-1R	MP-16S-<1R	MP-16S-2R
	1.00	4	MP-50S-8R	MP-65S-10R	MP-70S-30R	50-60S, 5-15R	60-80S, 10-40R	MP-40S-4R	MP-50S	MP-45S-6R
	2.00	8	MP-2S	MP-3S	MP-3S	0-5S	0-10S	MP-5S	MP-3S	MP-5S
Sulfamate Pb on sheet copper	0.25	3	100S(d)	100S(d)	100S(d)	0	0	30S(d)	50S(d)	30S(d)
	0.50	2	...	...	5S(d)	0	0	...	...	5S(d)
Fluoborate Pb on sheet copper	0.25	3	100S(d)	100S(d)	100S(d)	0	0	40S(d)	40S(d)	80S(d)
	0.50	3	...	...	3S(d)	0	0-10S(d)	...	...	3S(d)
Sulfamate Pb-Sn (2 per cent Sn) on steel	1.00	3	100R	MP-100S-50R	100R	0	0	90R	MP-100S-25R	90R
Sulfamate Pb-Sn (10 per cent Sn) on steel	1.00	3	100R	MP-100S-35R	100R	0	0	MP-50S-5R	MP-90S-1R	MP-97S-4R
Commercial hot-dipped terne (20 per cent Sn)	0.17	3	100R	100R	100R	0	0	100R	100R	100R
Commercial hot-dipped lead (2.5 per cent Sn, 2 per cent Sb)	0.40	3	100R	100R	100R	0	0	100R	100R	100R
Electroplated zinc on steel	0.20	3	100R	100R	100R	0	0	70R	90R	100R
Hot dipped zinc on steel	0.75	3	...	1R	MP-5S	0	0	...	...	...
Bare steel	...	4	100R	100R	100R	0	0	100R	100R	100R
Fluoborate Pb on steel (damaged)	0.50	3	100R	100R	100R			100R	100R	100R
	1.0	3	Bad Spread	Bad Spread	Bad Spread			MP-20S-3R	MP-20S-2R	MP-35S-2R
Fluoborate Pb on Cu-plated steel (damaged)	0.50	3	Very Bad Spread	Very Bad Spread	Bad Spread			MP-95S-63R	MP-100S-70R	100R
	1.0	3	Spread	Spread	Spread			MP-16S-<1R	MP-30S	MP-25S-1R

to tie in with previous exposure data, the Watt's nickel only is to be buffed. It will be seen with a special reference to lot Nos. 2 to 5 inclusive that this set of panels will particularly develop the effect of the thickness of the cyanide copper strike underlying the acid copper plate. The other 3 of the 7 lots are largely for purpose of reference only.

Each lot will consist of 24 panels of which 4 at each exposure site will be continuously exposed and rated with no cleaning; 4 other panels from each lot will be exposed at each site and regularly cleaned once per month. The cleaning procedure and the rating procedure will be determined prior to exposure by Section D of the subcommittee. The three exposure sites chosen for the test are Kure Beach (sea coast atmosphere), New York (industrial atmosphere), Detroit (relatively heavy industrial atmosphere). The regular cleaning of part of the panels is believed to be a pertinent addition to the test, it being suspected that products of corrosion remaining on the surface of the panels accelerates additional corrosion beyond that expected to be encountered in normal service.

It is anticipated also that the usual inspection will be supplemented by a laboratory investigation of selected panels to determine from time to time the extent of corrosion with respect to composite coatings.

It is desirable in this report to refer again to the data given in Table II where it will be noted that the ratings given at the end of 2.4 yr in New York are higher than those assigned at the end of 1.4 yr at the same location. It will be noted that this observation does not hold true at the Kure Beach site. It may be mentioned that the 1952 inspection in New York occurred immediately after several heavy rainfalls. This also may have contributed to a better surface appearance. More importantly, it is pertinent to mention that the ratings in New York at the end of 1.4-yr exposure gave considerable weight to surface pitting of the electrodeposited coatings themselves; whereas a year later this effect was de-emphasized, and, although some attention was given it, a greater weight was placed on the protective value offered the base metal by the plated coatings. It was noticed in New York that the industrial atmosphere caused a considerable amount of surface pitting on all panels including even wrought nickel which had been nickel and chromium plated. In view of this, the inspection group in 1952 agreed that the rating numbers should not be materially influenced by this effect. No such corrosion was noticed to any marked degree on any of the panels on exposure at Kure Beach. Therefore, from the standpoint of judging the real protective value of the coatings, more weight should be given to the ratings at the end of 2.4 yr in New York than those at the end of 1.4 yr at the same location.

ATMOSPHERIC EXPOSURE OF ELECTROPLATED LEAD COATINGS ON STEEL

Subcommittee II has continued the exposure tests on lead coatings and Table V is a continuation of the data last reported in 1949. The table covers inspections in 1949, 1950, and 1951. The panels at Tela, Honduras, were not inspected for this period. However, all panels including those at Tela have been inspected for the year 1952, or approximately 8.5 yr of exposure, and the results will be reported in 1954 along with weight loss data.

Inspection and comparison of the data will show that the trends previously reported are continuing. As last reported, from visual inspection, the most severe atmosphere is at Kure Beach and the least severe is at New York. The retarding effect of a copper undercoating on the rate of rusting is still apparent. While a 0.015-mil copper undercoat appears to aid corrosion protection of steel for lead coating thicknesses of 0.25 mil and greater, early results on the 0.08-mil lead deposits showed the copper undercoating to be harmful to corrosion protection in many cases.

Included in the table are data showing the range of visual inspection readings for the number of panels inspected. It is seen that in many cases there is considerable variation in the per cent stain and rust reported for panels of the same nominal coating. This is not only due to actual differences in corrosion but also to the inability of accurately reading the panels. For this reason, although doing so will leave some of the groups rather short in number of panels, it was decided by the subcommittee that panels would again be removed for lead weight loss, steel weight loss, and loss in tensile strength. These panels have been removed and the data will be reported in 1954.

METHODS OF RATING

Because of several criticisms that the method used by Subcommittee II for rating decorative chromium-plated panels was not adequate and frequently misinterpreted, Section D was formed. The subcommittee's instructions were to review suggestions made and make recommendations for overcoming the deficiencies of the method and to simplify evaluation of results.

The present method[3] of rating decorative panels consists of assigning numbers denoting the total area defective and describing the types of defects by means of a shorthand code. The rating number is assigned by comparing the panels to photographs and charts. While this system of rating panels is basically sound, many people when reading the report of Subcommittee II are interested only in the numerical value assigned to a panel or group of panels and evaluate the results of the test on the number alone. In many instances panels with equal areas defective are given the same rating number even though one of the panels may not be corroded as severely as the others and would be considered to be in better condition. The shorthand code description, which few people read and study, is intended to show this difference.

Another deficiency of the method of rating used by Subcommittee II was that the ratings were assigned to a group of panels representative of the variable being studied, rather than to individual panels. This did not permit statistical analysis of the results.

A third deficiency of the method was that in many instances groups of panels with noticeable differences were given the same rating number. One group would have the maximum amount of area defective for that number, while the other group would have the minimum amount of area defective.

In studying the suggestions made, it was evident that a method of rating panels in which the types of defects are weighted was desired. In addition, the section realized that the weighting given to the various types of defects would vary with individuals and their use of the data.

The following rating procedure is proposed for the rating and the presentation of data for the evaluation of protection by decorative plating on test panels exposed to the atmosphere on stationary exposure racks. It is recommended specifically for the Performance Test Program No. 3 of copper-nickel-chromium plated steel panels. The panels which are to be cleaned periodically are to be rated separately from the panels which are to be exposed continuously without periodic cleaning.

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[3] W. A. Wesley, "Rating Exposure Test Panels of Decorative Electrodeposited Cathodic Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 803 (1947); Report of Subcommittee II on Performance Tests, *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 220 (1949).

Section D recognizes that the proposed rating procedure will be more time consuming than the procedure previously used. However, in view of the time consumed and money spent in planning programs and in preparing test specimens, Section D believes that the additional time required is very small when compared to the information gained.

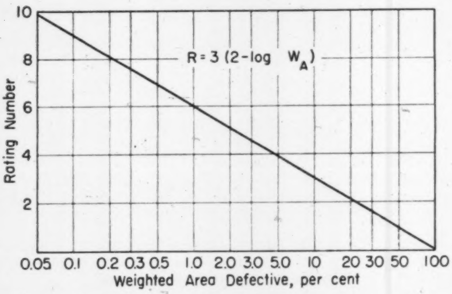

FIG. 1.—Chart for Converting per cent Area Defective to Rating Number.

A. *Panels not to be cleaned periodically:*

1. *Arrangement on Racks.*—At the first inspection and each subsequent inspection, the panels are to be arranged on the test racks in order of decreasing commercial value from the standpoint of appearance. Duplicate panels with various degrees of failure will probably not be adjacent to each other on the racks and may be separated by several other panels. The first inspection will require the greatest amount of rearrangement on the racks. Subsequent inspections may require some rearrangement, but it is not expected to be seriously time consuming.

2. *Assignment of Rating Numbers.*—With the aid of photographs or charts (Fig. 1) now used by Subcommittee II, the per cent area defective for each type of defect is to be estimated and recorded. This can be done by finding the panels with the per cent area defective closest to those shown in the photographs or charts. Panels showing intermediate degrees of attack can then be estimated by comparing them to the panels previously rated. Before leaving the test site, arbitrarily assign a weighting factor to each type of defect in accordance with the inspector's judgment of the seriousness of the defect compared to pinhole rusting. Use this factor to find the final weighted area for each defect.

Thus

$$fa = W_a$$

where:

f = factor or weighting,
a = per cent area defective for any given type of defect, and
W_a = final weighted area. Then after each type of defect is rated, the several weighted areas are added together to give the total weighted area.

Thus

$$\Sigma W_a = W_A$$

where:

W_A = the total weighted area. Then by using the formula (or referring to the attached curve)

$$\text{Rating} = 3(2 - \log_{10} W_A)$$

the total weighted area is converted to the proper rating. By using this formula a rating of 9 would approximately equal 0.10 per cent area defective. A rating of 8 would approximately equal 0.25 per cent, etc. A rating of 10 would be assigned to a panel with a total weighted defective area of less than 0.05 per cent. A rating number of 0 would be assigned to a panel with a total weighted defective area greater than 100 per cent. Results reported should include the defective area percentages for each type of defect, as well as the weighted rating number for each panel. After assigning the rating numbers for all panels in a group, they should be reinspected to determine if an error has been made in arranging the panels, in estimating per cent area defective, or in assigning weighting factors. These errors will be apparent where a rating number indicates that a panel is out of order when compared to adjacent panels.

When deemed advisable by the inspectors, the metals undergoing corrosion may be detected by various chemical tests. The following tests are suggested and should be applied to small areas which are being corroded. Only one of duplicate panels shall be tested in this manner. The results of these tests will assist in determining the defects being rated and in determining the weighting factors.

Nickel: Saturate filter paper with NH_4OH or distilled water. Place filter paper on panel and rub lightly. Remove filter paper and add dimethyl-glyoxime. Red precipitate indicates presence of Ni.

Iron: Saturate filter paper with dilute HNO_3. Place filter paper on panel and rub lightly. Remove paper and add drop of $K_4Fe(CN)_6$. Blue color indicates presence of Fe. (If no iron or extremely small amounts are present, green or faint purple color may appear. Disregard this).

Copper: Saturate filter paper with dilute HNO_3 or with distilled water. Place filter paper on panel and rub lightly. Remove filter paper and add drop of $Zn(C_2H_3O_2)_2$ to filter. Paper followed by drop of $(NH_4)_2Hg(CNS)_4$. Purple or light pink color, depending on the amount of Cu present, indicates the presence of copper.

NOTE.—Iron will cause a reddish precipitate to appear. The iron may be masked out by adding an oxalate, but usually the test will show a positive purple color even with fairly large amounts of iron present.

Solutions:

1. HNO_3—dilute 1:5
2. NH_4OH—dilute 1:1
3. Dimethylglyoxime—1 per cent in alcohol
4. $K_4Ke(CN)_6$—10–15 per cent solution
5. $Zn(C_2H_3O_2)_2$—1 per cent
6. $\left.\begin{array}{l} NH_4CNS\text{—9 g} \\ HGCl_2\text{—8 g} \end{array}\right\}$ 100 ml H_2O

B. *Panels to be cleaned periodically:*

1. Section D recommends that the panels which are to be cleaned periodically be cleaned once each month by the following procedure. Remove loose dirt by rinsing with water and wiping with a wet cloth. Remove surface corrosion products by rubbing with a water paste of Johns Manville's Super Floss (2 to 4 micron diatomaceous earth), allow paste to dry, and remove with a soft dry cloth.

2. Inspection of the panels cleaned periodically should coincide if possible with scheduled cleaning. Without rearranging panels on racks rate test panels as outlined under A-2 after removing the loose dirt by rinsing with water and wiping with a wet cloth.

3. Remove corrosion products by rubbing with a water paste of a mild abrasive such as Johns Manville's Super Floss. Allow paste to dry and remove with a soft dry cloth.

4. Arrange panels on racks in order of decreasing commercial value from the standpoint of appearance.

5. Rate test panels again as outlined in paragraph A-2.

The factors for weighting each type of defect may be changed after calculating rating number and reinspecting panels. As a preliminary guide, the following factors are suggested:

Pinhole rusting	1
Light irridescent stain	1/100
Crater rusting	4
Blisters	2
Peeling and flaking	4
Light surface pits	1/100
Crows feet and moderate to heavy surface pitting	1/4
Dark stain	1/10

Respectfully submitted on behalf of the subcommittee,

W. L. PINNER,
Chairman.

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1952, to June, 1953. During this time, one meeting was held in Cleveland, Ohio, on April 22, 1953.

During the year, W. A. Reich, chairman of Committee B-9 since its organization in 1944, resigned the chairmanship because of change in employment. The Advisory Committee appointed F. V. Lenel to serve out W. A. Reich's unexpired term, and C. G. Johnson to serve as secretary in place of F. V. Lenel.

NEW TENTATIVE

The committee recommends to the Society the publication as tentative of the proposed Tentative Specifications for Sintered Metal Powder Structural Parts from Brass appended hereto.[1] This proposed tentative has been submitted to letter ballot of the committee, and the results will be announced at the Annual Meeting of the Society.[2]

EDITORIAL REVISION OF TENTATIVE

The committee recommends to the Society the following revision to the Tentative Recommended Practice for Evaluating the Microstructure of Apparent Porosity in Cemented Carbides (B 276 - 52 T).[3] The title of this tentative is to be changed to "Tentative Recommended Practice for Evaluating Cemented Carbides for Apparent Porosity." This revision has been submitted to letter ballot of the committee, and the results will be announced at the Annual Meeting of the Society.[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specification for Metal Powder Sintered Bearings (B 202 - 51 T)[2], Tentative Definitions of Terms used in Powder Metallurgy (B 243 - 49 T)[2], and Tentative Specification for Sintered Metal Powder Structural Parts from Bronze (B 255 - 51 T)[2] be retained as tentative in view of several pending and proposed additions and changes in each of these tentatives.

ADVISORY COMMITTEE ACTIVITIES

The Advisory Committee held three meetings during the year to act on various committee administrative matters. New chairmen were appointed for three of the sections of Committee B-9. Through the efforts of the committee, the consumer and general interest representation on the committee was enlarged during the year.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman).—Further consideration was given to additional terms for inclusion in the Glossary (Definitions of Terms Used in Powder Metallurgy, B 243 - 49 T). The noun "Sintering," describing a product made by sintering metal powders, first considered by the subcommittee las year, has found wide application during the past year. It was decided to submit whether this term should be incorporated in the Glossary to a letter ballot of Committee B-9. The work on technical data sheets on material covered by Specification for Sintered Metal Powder Structural Parts (B 222 - 52) is being continued.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29-July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to 1952 Book of ASTM Standards, Part 2.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

[3] 1952 Book of ASTM Standards, Part 2.

Subcommittee II on Metal Powders (J. J. Cordiano, chairman).—Minor changes were made in the four tentative methods for Chemical Analysis of Metal Powders mentioned in the 1951 report. These tentatives are being submitted to letter ballot of the committee and simultaneously forwarded to Committee E-3 on Chemical Analysis of Metals with the hope that they can be included in the next issue of the ASTM Volume on Methods of Chemical Analysis of Metals. The tentative on Subsieve Particle Size Analysis is also being submitted to committee letter ballot. Efforts to standardize on a compressibility test are continuing, and work on a test for green strength is being taken up.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman).

Section A on Bearings (Paul Moench, chairman) agreed on a recommended size list for porous bearings which is to be appended to Tentative Specifications for Metal Powder Sintered Bearings (B 202 - 51 T). Several other minor changes in B 202 were also decided upon. Both the size list and the other changes are to be submitted to letter ballot of the section.

Section B on Structural Parts (P. J. Shipe, chairman) held two meetings during the year. The Specifications for Brass Structural Parts were completed, approved by both the section and Committee B-9, and are appended to this report.[1] Substantial agreement was reached on Tentative Specifications for Copper Impregnated Structural Parts, which will be submitted to letter ballot of the section.

Section C on Cemented Carbides (R. A. Canning, chairman) continued its efforts in developing standards for testing cemented carbides with a standard for hardness testing being submitted to letter ballot of the section and procedures for transverse rupture testing and specific gravity determination being drafted for further discussion. Two new task forces on metallographic procedures for cemented carbides and on classification of cemented carbides are being appointed by the chairman of the section.

This report has been submitted to letter ballot of the committee, which consists of 58 members; 30 returned their ballots, of whom 20 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. V. LENEL,
Chairman.

C. G. JOHNSON,
Secretary.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION*

The 1952 report[1] of the Advisory Committee on Corrosion summarized the activities of the technical committees of the Society engaged in the study of exposure testing. There seems little point in making such complete reviews annually, but this information will be brought up to date periodically. There are, however, a number of new or continuing tests which warrant brief mention.

EXPOSURE PROGRAMS

Committee A-5 on Corrosion of Iron and Steel:

Subcommittee XIV on Sheet Tests now has specimens only at Annapolis, Md. (copper-bearing and non-copper-bearing sheets since 1916) and Altoona, Pa., and State College, Pa. (uncoated and galvanized sheets since 1926).

Subcommittee XV on Wire Tests is reporting the results of the past two years' annual inspections in its biennial appendix to the 1953 Report of A-5.[2] Present sites include Bridgeport, Conn., State College, Pa., Lafayette, Ind., Ames, Iowa, Manhattan, Kan., Ithaca, N. Y., Santa Cruz, Calif., College Station, Tex., Davis, Calif., and Pittsburgh (Williams & Co.), Pa.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Subcommittee VII on Weather has continued the exposure of iron and of zinc specimens for the purpose of calibrating the relative corrosivity of various test site locations. Specimens have been exposed during the past year at State College, Pa., New York City, Point Reyes, Calif., Kure Beach, N. C., and Pittsburgh, Pa. An interim report is to be presented by C. P. Larrabee and O. B. Ellis at the June, 1953, meeting of the Society.[3]

Subcommittee VIII on Galvanic and Electrolytic Corrosion has completed the exposure of Part 2 of its 3-part program on galvanic magnesium couples. Part 2, wire-wound bolts, consisted of three sets of specimens exposed for three consecutive four-month periods and a fourth set exposed over a total period of 12 months. The first of three series of disk-type couples (Part 1) was removed last year. The materials for the plate-type couples of Part 3 are currently being assembled.

Committee B-6 on Die-Cast Metals and Alloys:

Subcommittee V on Exposure and Corrosion Tests, which some years ago exposed a number of die-cast aluminum alloys, is currently making additional exposures of the test specimens which had been held in reserve. These exposures are being made at Kure Beach, New York City, and Columbus, Ohio.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Subcommittee VIII on Atmospheric Exposure Tests has been engaged in the exposure of more than 1500 (per site) aluminum and magnesium specimens at New York City, State College, and Kure Beach. Additional exposures are planned for Point Reyes, Calif., and Freeport, Tex.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Report of Advisory Committee on Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 212 (1952).

[2] See p. 101.

[3] See p. 194.

Details will be given in a paper by L. H. Adam on "Resistance of Light Metals to Atmospheric Exposure," at the June, 1953, meeting of the Society.[4]

Committee B-8 on Electrodeposited Metallic Coatings:

Subcommittee II on Performance Tests is continuing its study on copper-nickel-chromium and lead deposits on steel as well as currently planning tests for the purpose of ascertaining the relative protection afforded by various supplemental organic coatings. An additional series of plated panels will be exposed within the next few months and panels with supplemental organic treatments are planned for exposure by the end of the year.

TEST SITE ENVIRONMENTS

It is hard to realize from a verbal description the appearance of the various sites as well as the environment surrounding them. In an effort to make this information available to those interested, photographs of the various ASTM test sites have been incorporated in an Appendix to this report.[5] These photographs have been chosen to show the various test sites and their surroundings rather than the details of the types of specimens.

This report has been submitted to letter ballot of the committee, which consists of 28 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. L. LAQUE,
Chairman.

J. S. PETTIBONE,
Secretary.

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[4] See p. 227.

[5] See p. 274.

APPENDIX

ASTM TEST SITES

The American Society for Testing Materials, with authority vested in its Advisory Committee on Corrosion, now has under its jurisdiction 23 atmospheric test sites representing marine, industrial, rural, and tropical atmospheres. The marine atmospheres include East, West, and Gulf Coasts. Industrial atmospheres range from the highly industrialized Pittsburgh area to semi-industrial locations such as Columbus, Ohio. Rural atmospheres range across the United States from Santa Cruz, Calif., in the west, College Station, Tex., in the south, Ames, Iowa, in the central, and Ithaca, N. Y., and State College, Pa., in the northeast sections The tropical exposures are mild in Miraflores, C. Z., and severe at Fort Sherman, C. Z.

The industrial test site at *Altoona, Pa.* (Fig. 1), is located on the fourth floor roof of the Pennsylvania Railroad's General Office and Storehouse Building of the Juniata Shops. Adjacent to the Juniata Shops the Pennsylvania Railroad has a large classification yard, and the specimens in this test site are subjected to extensive fumes from both coal burning and diesel powered locomotives.

The rural sites of *Ames, Iowa* (Fig. 2), and *Annapolis, Md.* (Fig. 3), were set up for the A-5 wire tests and sheet tests, respectively. The Ames site is located on the grounds of the Iowa State College but is located some distance from any of the college buildings. The Annapolis site, located several hundred yards from the Severn River is located within the U. S. Naval Engineering Experiment Station and as can be noted from the photograph these specimens are, during certain hours of the day, shaded by nearby trees.

One of the industrial sites used by A-5 in connection with its wire tests is located in *Bridgeport, Conn.* (Fig. 4). To accommodate the owners of the plot, the area is to be reduced to one half its present size.

College Station, Tex. (Fig. 5), representative of the rural south, was another of the sites chosen for the A-5 wire tests. This site is located on the grounds of the Texas Agricultural and Mechanical College.

Columbus, Ohio (Fig. 6), is a semi-industrial site picked so as to represent conditions intermediate between those prevailing in Pittsburgh and State College, Pa. The specimens are located on the fourth floor of the Seventh Avenue Building as indicated by the arrow in Fig. 6. There are railroad yards approximately 2½ miles southeast and the prevailing winds in this area are from that direction.

Davis, Calif., is the West Coast rural site used for the A-5 wire tests and is located on the University of California grounds This site is shown in Fig. 7.

The industrial site of *East Chicago, Ind.*, is located near typical industrial plants such as the iron foundry, gas works, etc., shown in the background of Fig. 8.

Through the courtesy of the U. S. Naval Research Laboratories an agreement has been reached whereby the NRL is willing to permit the ASTM limited usage of its test site facilities at *Fort Sherman* (also Miraflores, C. Z.) in the Canal Zone. These facilities are surrounded by tall trees and a heavy undergrowth, conditions characteristic of the hot humid type of environment found in the jungle. General views of this area are shown in Fig. 9.

The *Freeport, Tex.*, area is a Gulf Coast site and is located in the floodgate area at the mouth of the Brazos River. This is the latest site acquired by the Society and the area to be used is indicated by the flags and white outline in Fig. 10.

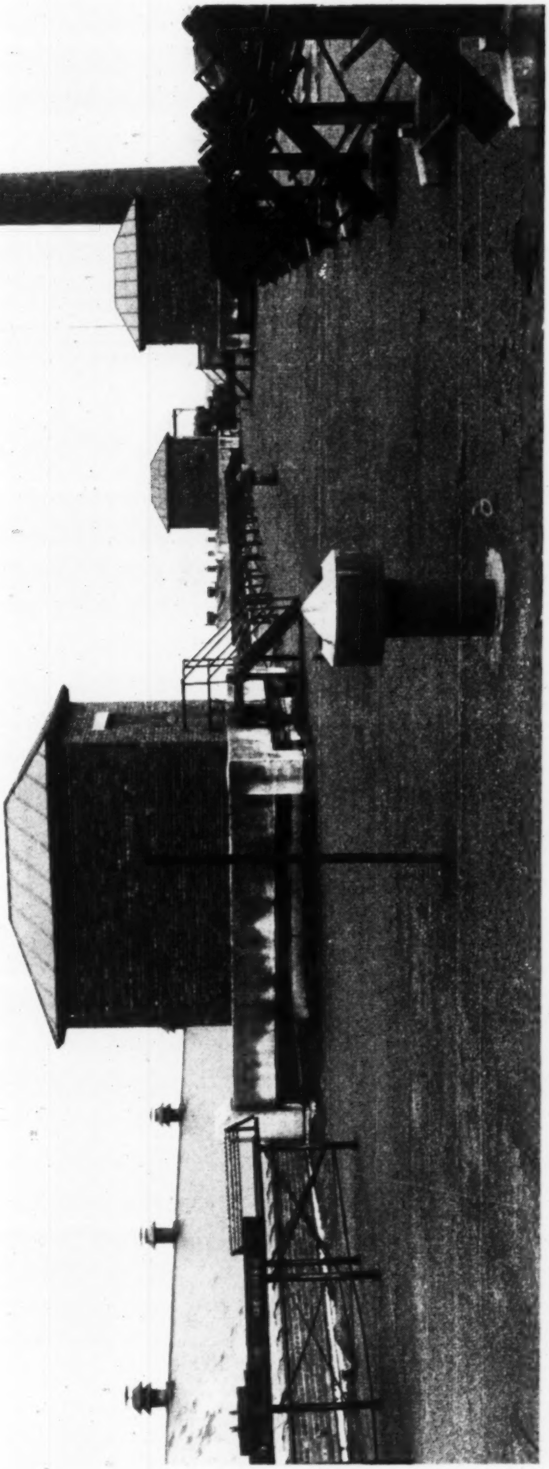


FIG. 1.—Altoona, Pa.

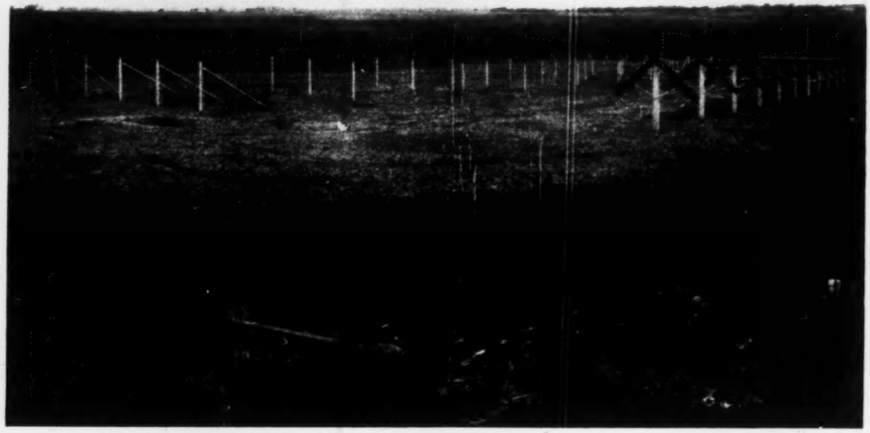


FIG. 2.—Ames, Iowa.

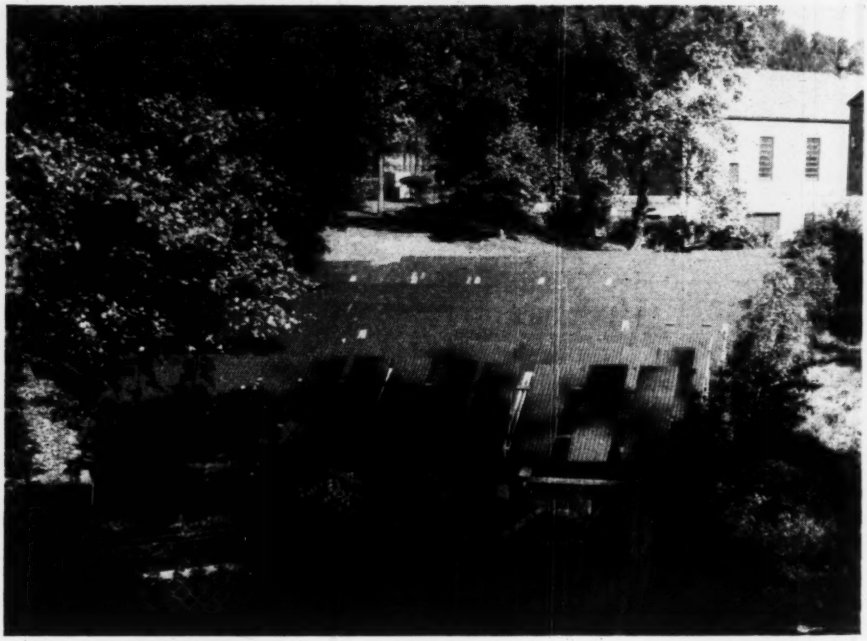


FIG. 3.—Annapolis, Md.

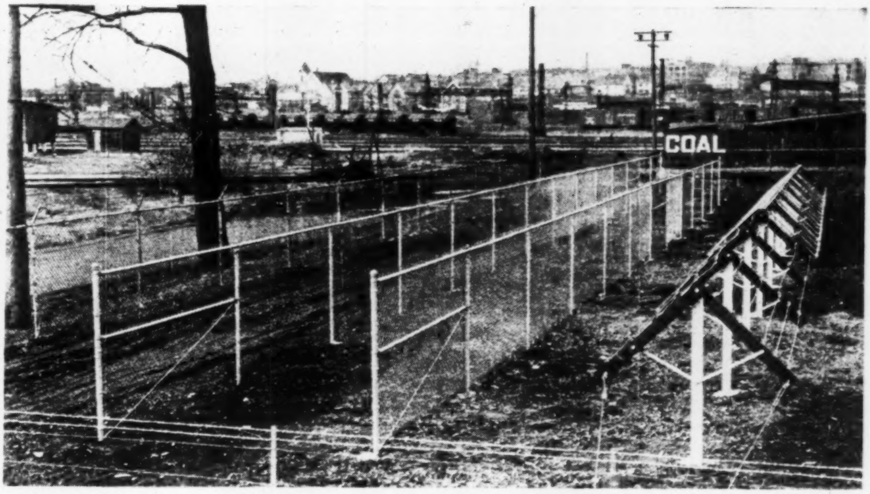


FIG. 4.—Bridgeport, Conn.

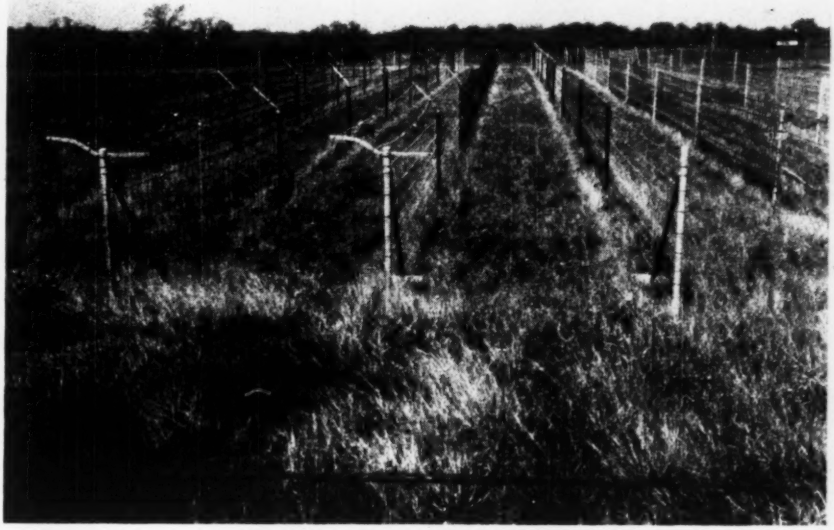


FIG. 5.—College Station, Tex.

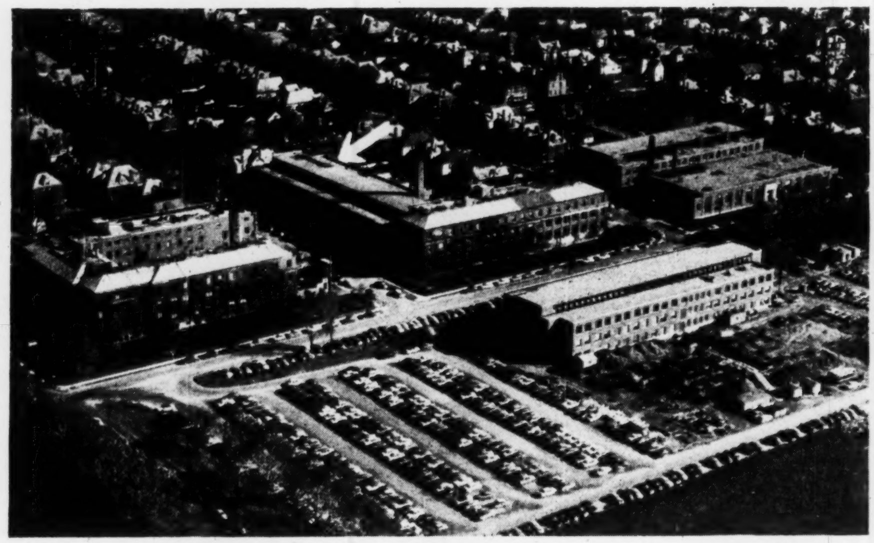


FIG. 6.—Columbus, Ohio.

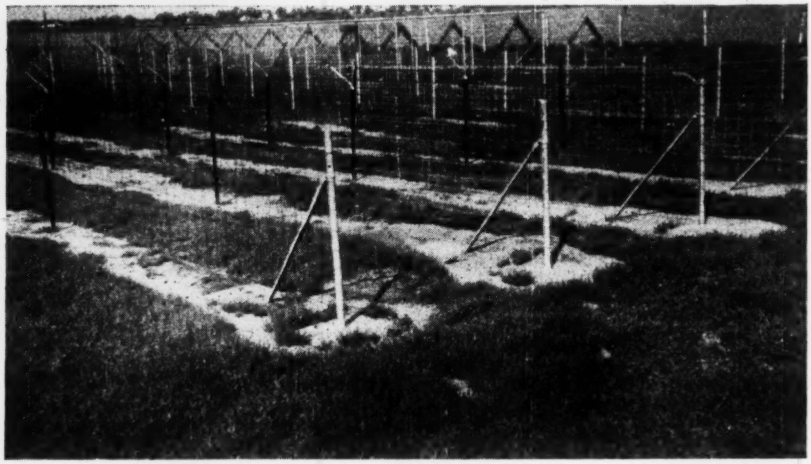


FIG. 7.—Davis, Calif.

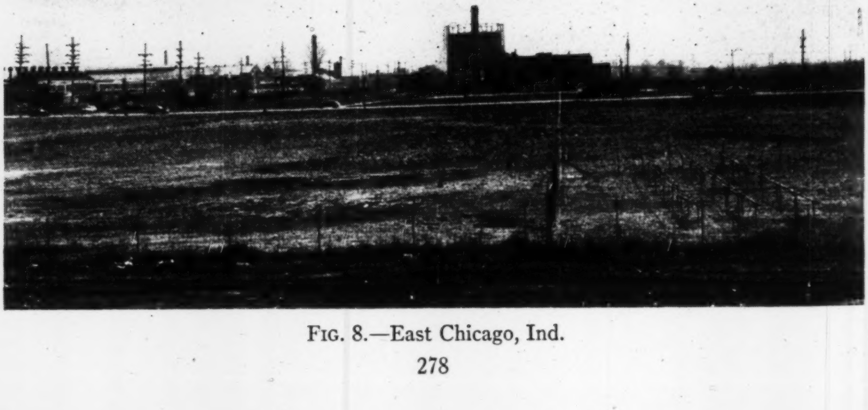
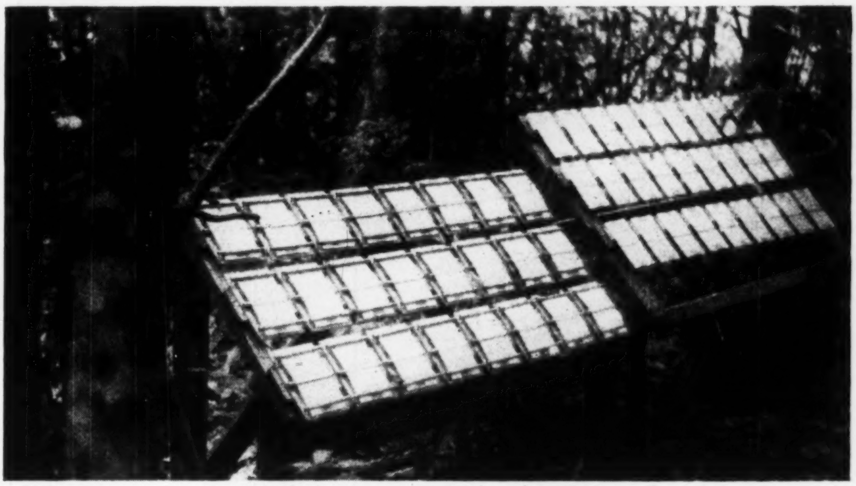


FIG. 8.—East Chicago, Ind.



(*a*) Specimens at Fort Sherman, C. Z.

(*b*) View of General Environment.

Fig. 9.—Fort Sherman, C. Z.

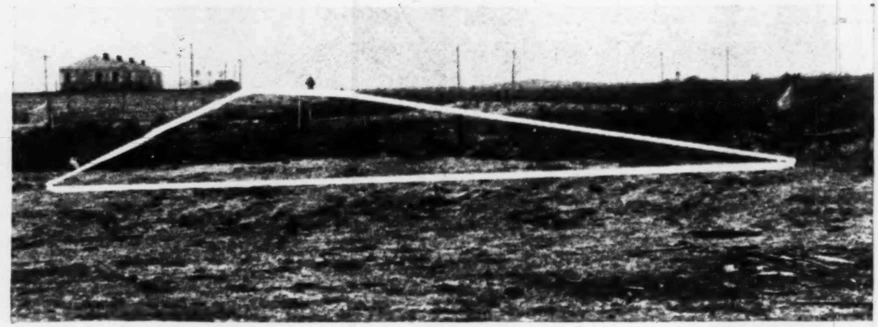


FIG. 10.—Freeport, Tex.

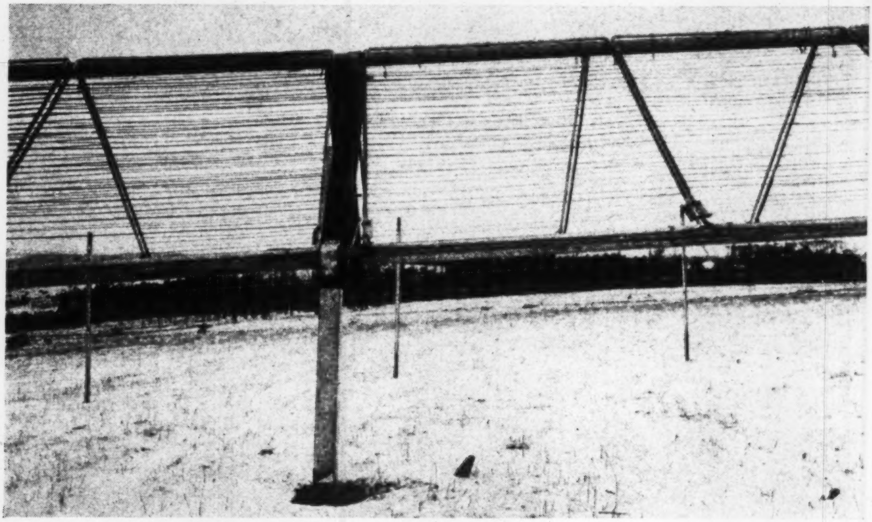


FIG. 11.—Ithaca, N. Y.

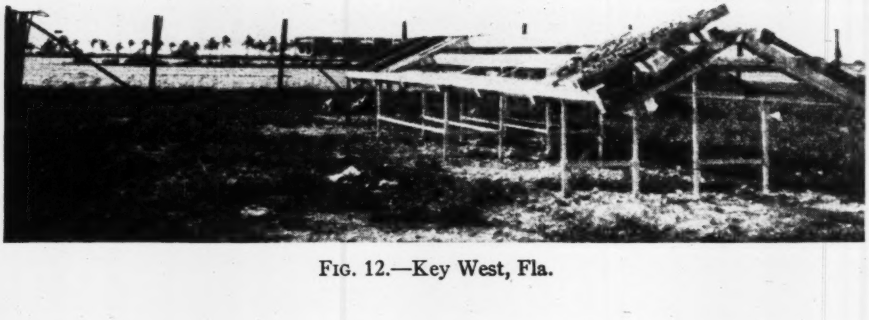


FIG. 12.—Key West, Fla.

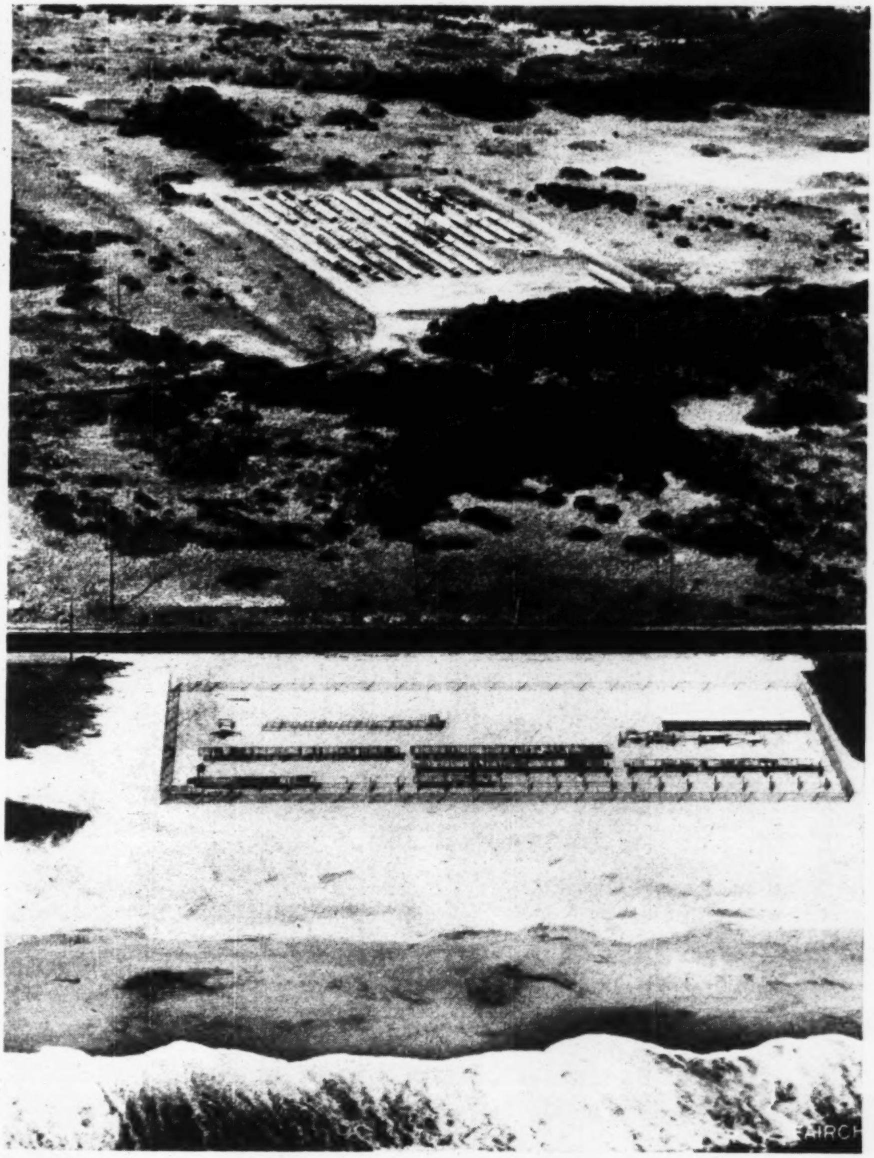


FIG. 13.—Kure Beach, N. C.

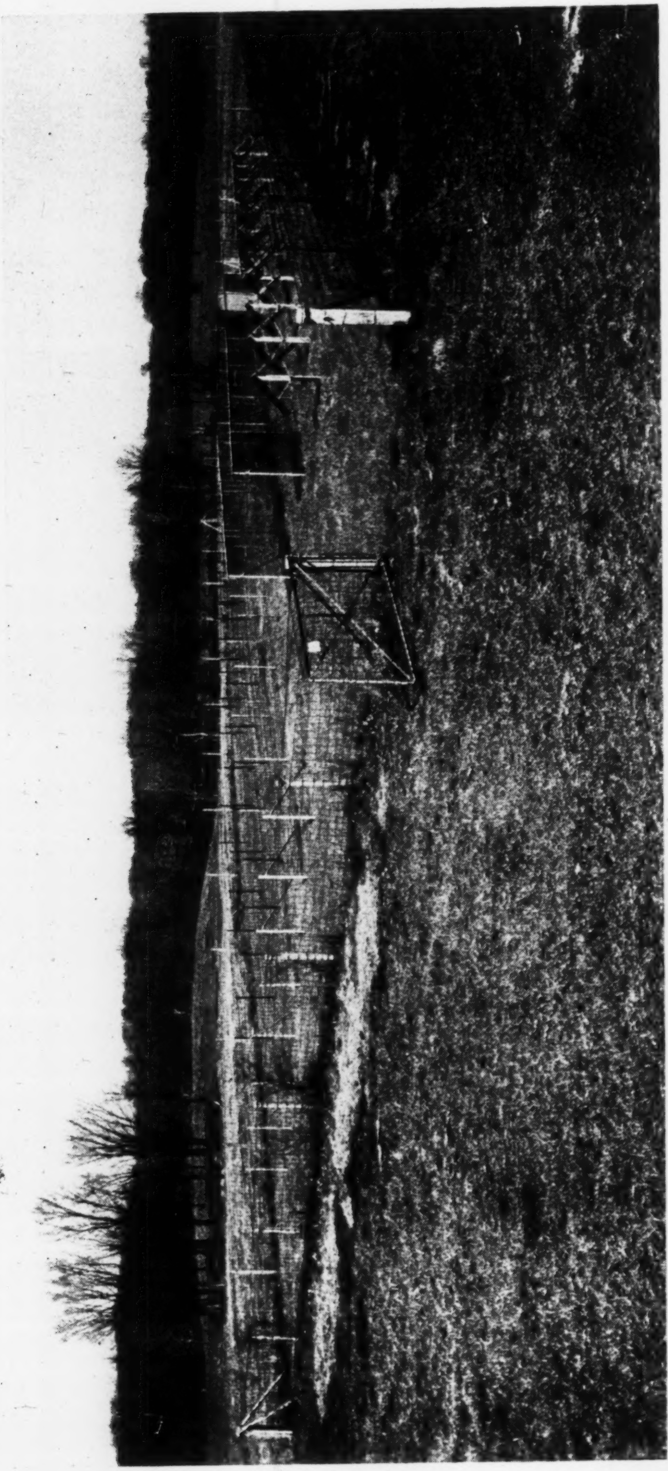


FIG. 14.—Lafayette, Ind.

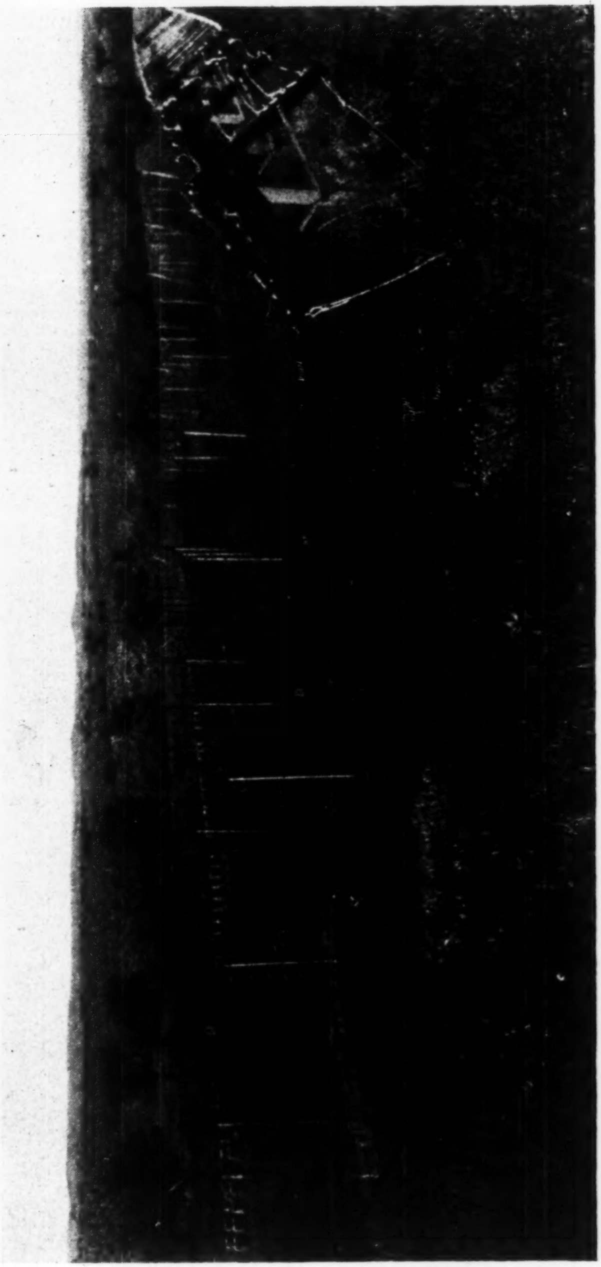


FIG. 15.—Manhattan, Kan.

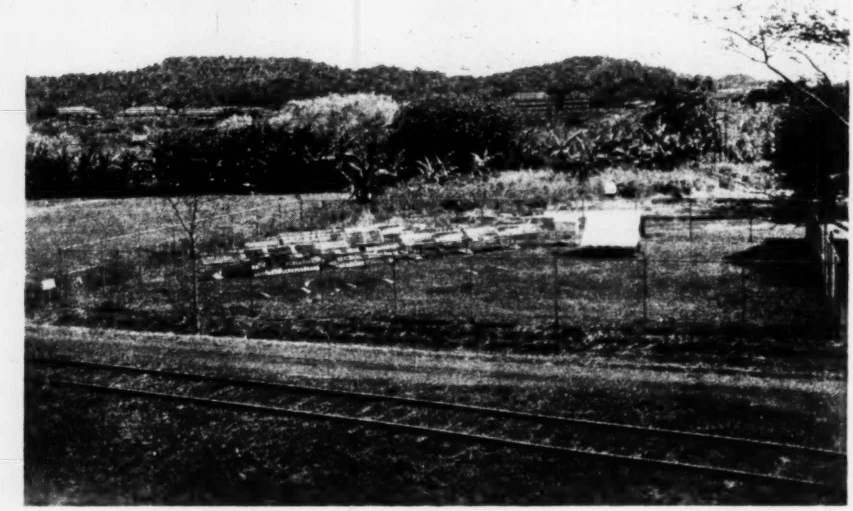


FIG. 16.—Miraflores, C. Z.

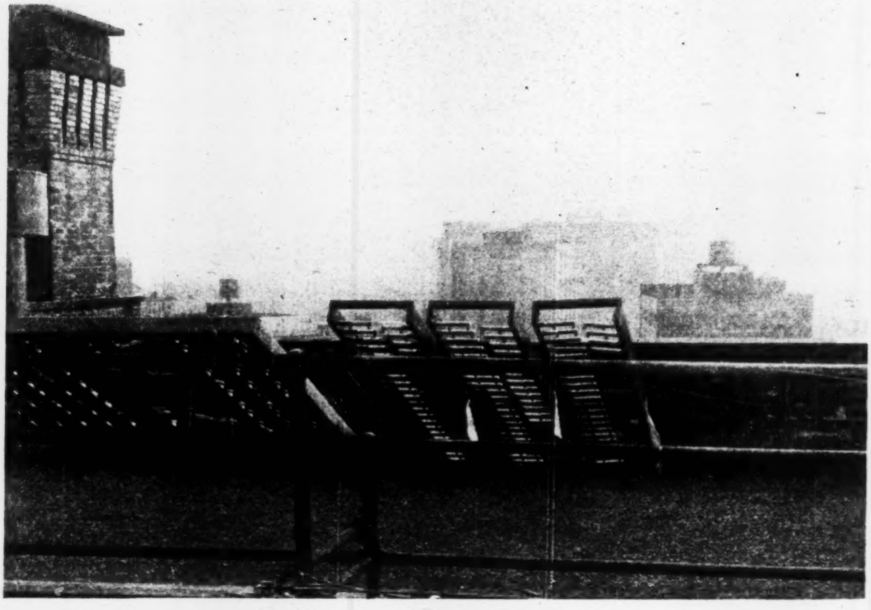
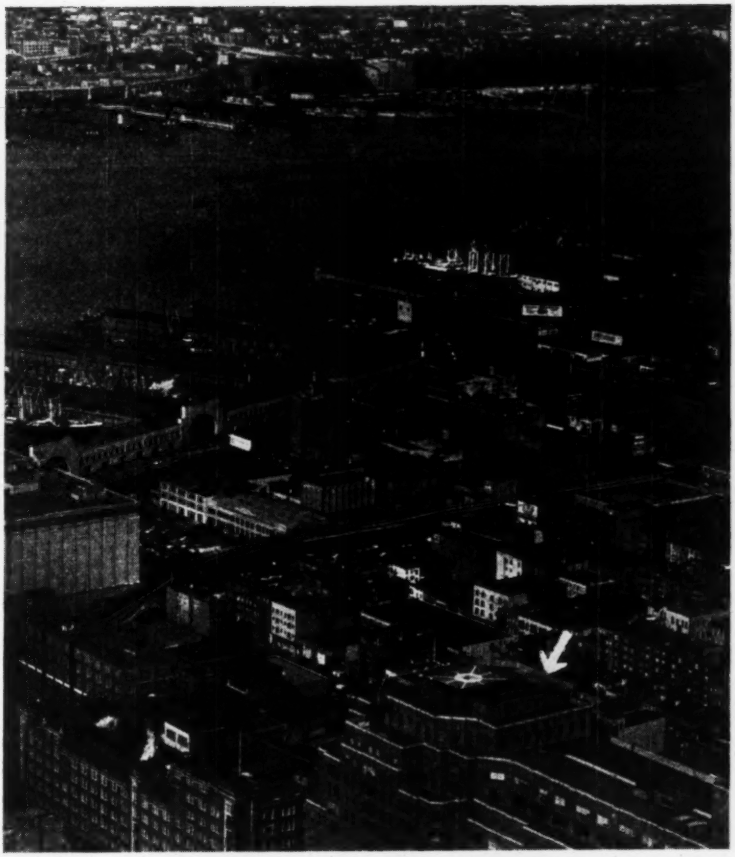
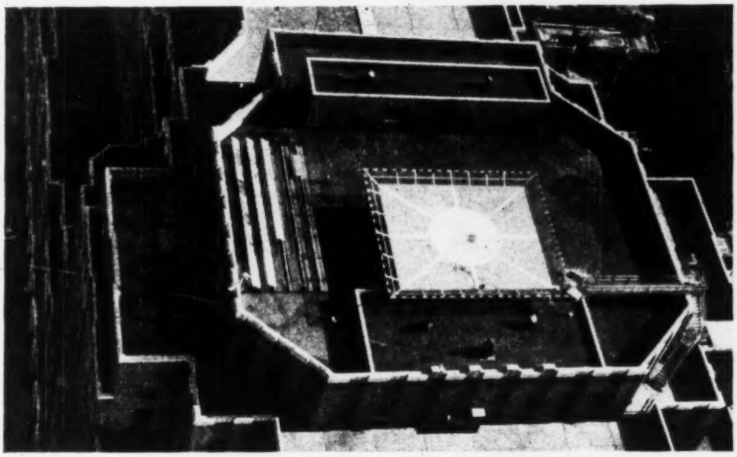


FIG. 17.—New York City (Bell Telephone Labs.).



(*a*) General View (looking northwest) of Manhattan Showing Port of New York Authority Building (white arrow, right foreground).



(*b*) Sixteenth Floor Penthouse Roof (looking east).

FIG. 18.—New York City (Port of New York Authority).

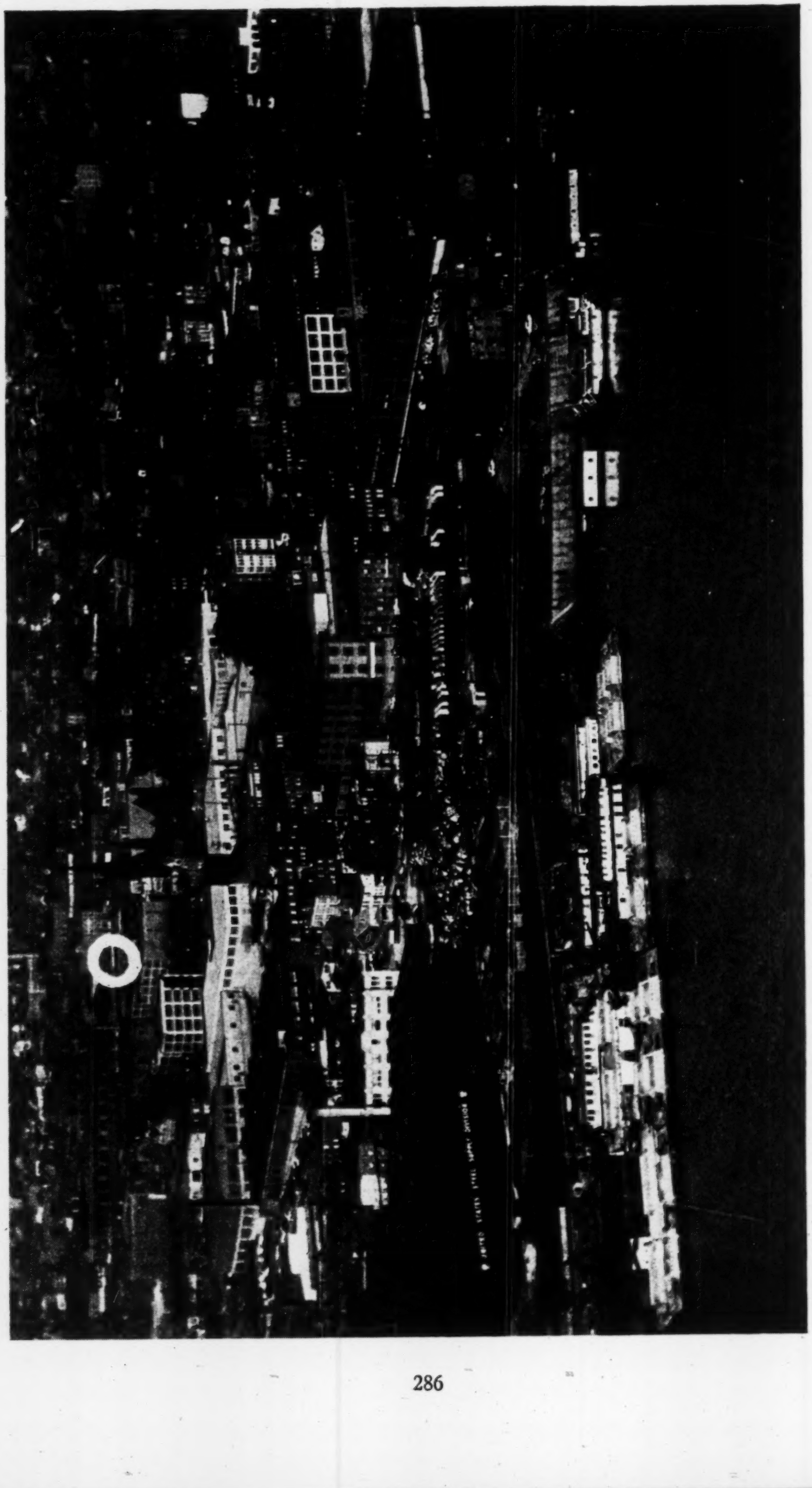


FIG. 19.—Pittsburgh, Pa.

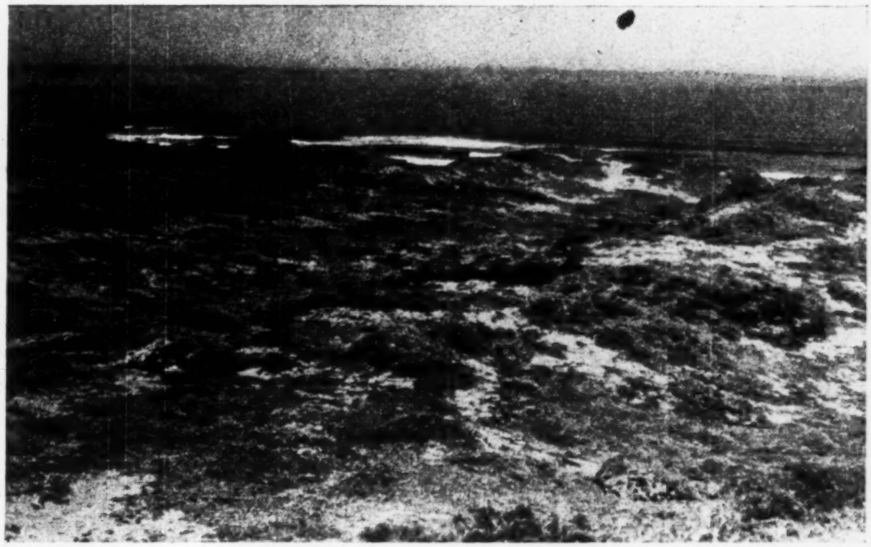


FIG. 20.—Point Reyes, Calif.

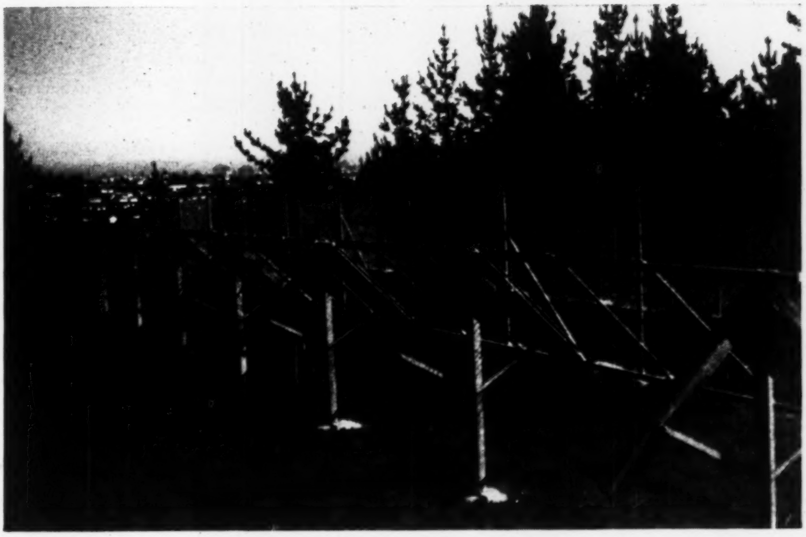


FIG. 21.—Santa Cruz, Calif.

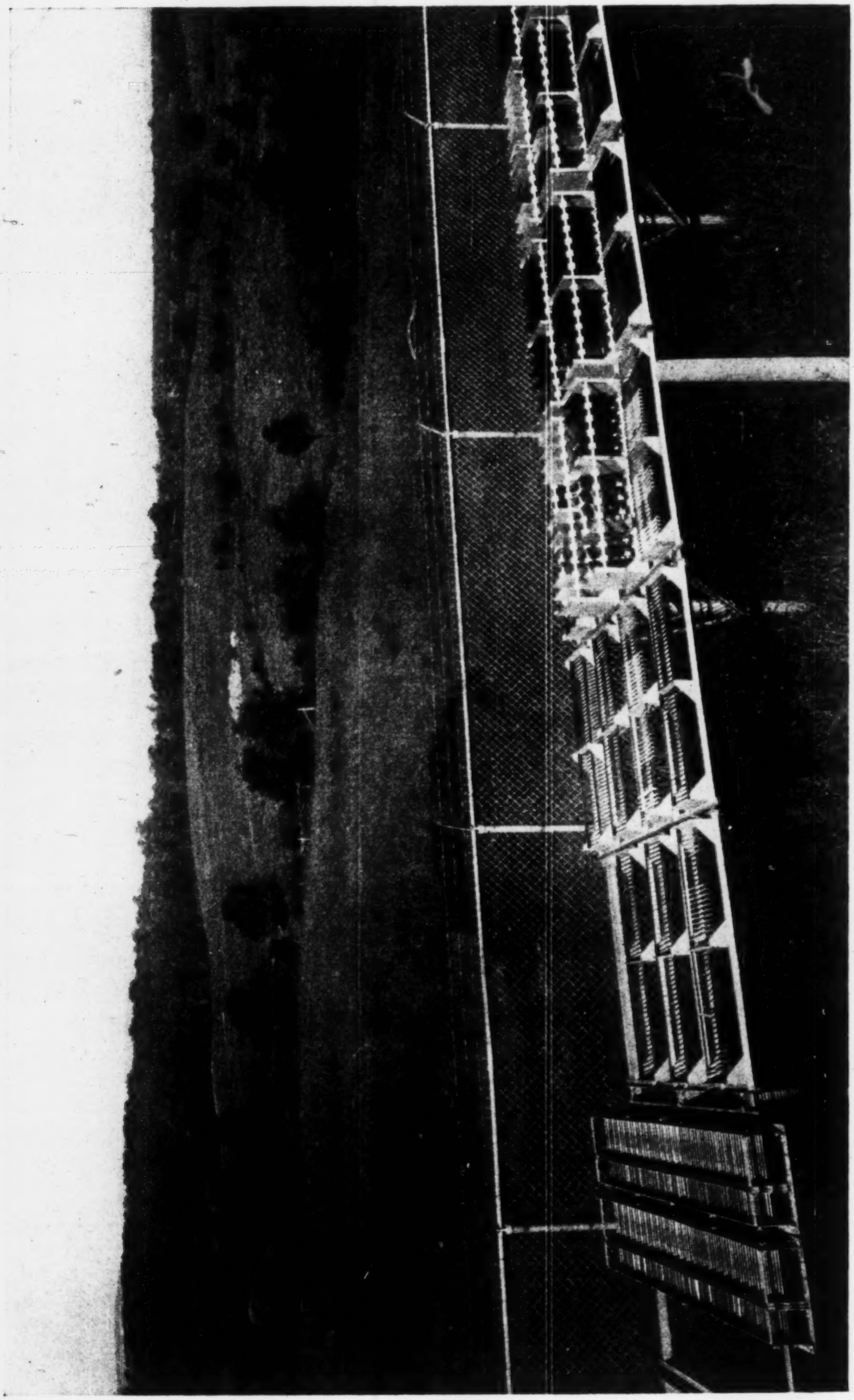


FIG. 22.—State College, Pa.

Ithaca, N. Y., is the northernmost site used by the Society and was set up at Cornell University by Committee A-5 for its wire tests. This area is shown in Fig. 11.

The *Key West, Fla.*, site, the southernmost site in continental United States, was recently relocated in the so-called salt ponds, a part of the U. S. Navy Reservation, and the specimens are presently located about one half mile from the Atlantic Ocean. The shore line is denoted by the palm trees in the background (Fig. 12). The site is about 6 ft above the surrounding brackish waters.

The *Kure Beach, N. C.*, location is the site of The International Nickel Co.'s exposure station and the meeting place of the well-known Sea Horse Inst. Figure 13 shows an aerial view of the 80-ft racks in the foreground and the 800-ft racks in the background.

Lafayette, Ind. (Fig. 14), and *Manhattan, Kan.* (Fig. 15), are representative of the rural areas of the Midwest and are located at Purdue University and Kansas State University, respectively.

Miraflores, C. Z., is not strictly an ASTM test site but space has been made available (also at Fort Sherman, C. Z.) to the Society through the courtesy of the Naval Research Laboratories. The Miraflores site is in an open area and is classified as mild tropic. A view of this area is shown in Fig. 16.

There are presently two sites in *New York City*, the earlier site being established on the roof of the *Bell Telephone Laboratories* in 1928 (Fig. 17). The newer site established on the sixteenth story penthouse roof of the *Port of New York Authority Building* is shown in two views in Fig. 18. The penthouse roof is shown in the general view of that area of Manhattan by the white arrow in the lower right-hand corner. The detailed view shows approximately 175 ft of pipe frames already in use.

FIG. 23.—Tucson, Ariz.

Various difficulties have been encountered in the *Pittsburgh, Pa.*, area. After many years the test site on Brunot Island had to be discontinued in 1951, and selected specimens were moved to a location on the roof of the Bureau of Mines. Last year it was found necessary to again relocate the test site and the present specimens consisting only of a few selected A-5 wires and the calibration specimens of B-3, Subcommittee VII, are currently located on the roof of the Williams & Company warehouse. The white circle in the upper left-hand section of Fig. 19 indicates the location of Williams & Company.

Although Fig. 20 does not show the facilities available at *Point Reyes, Calif.*, it does indicate the general terrain. The ocean can be seen in the background as viewed from a slight elevation just to the west of the ASTM site. The plot is approximately one half mile from the water.

Santa Cruz, Calif., the West Coast marine site used for the A-5 wire tests, is on land owned by the City of Santa Cruz and, as shown in Fig. 21, is located on a hill overlooking the city and Monterey Bay (left background).

State College, Pa., is quite typical of the rural areas of our country. This site, established in 1925, has been expanded and there is currently available somewhat more than half an acre of ground. A few of the several thousand specimens exposed at State College are shown in the foreground of Fig. 22. The area beyond the fence is gently rolling and typical of the area surrounding the site. There is no industry of any kind in the area.

As opposed to the rural type atmosphere characterized by State College, Pa., it was agreed that a hot, dry, rural area should be picked as a test site in the Southwestern part of the United States. Such an area has been made available by the U. S. Dept. of Agriculture through the Southwestern Forest and Range Experiment Station located at *Tucson, Ariz.* This area is located at the foot of a gentle slope as shown in Fig. 23, and the area proposed for the ASTM site is located about where the man is standing in the foreground.

Acknowledgment:

The committee acknowledges with thanks the assistance which was rendered by the following in connection with the procurement of the necessary photographs:

V. E. Amspacher, H. T. Cover, and M. A. Pinney, Pennsylvania Railroad;
Henry Giese, Iowa State College;
J. L. Thompson, U. S. Naval Engineering Experiment Station;
C. M. Shigley and W. G. Dudley, Dow Chemical Co.;
Lt. Col. L. W. Gross, Corps of Engineers;
C. H. Lucke, American Chain and Cable Co.;
H. P. Smith, Texas A & M College;
W. A. Welcker, Jr., and H. A. Pray, Battelle Memorial Institute;
L. W. Neubauer, University of California;
W. Bonsack, Magnesium Company of America;
F. L. LaQue, The International Nickel Co.;
I. D. Mayer, Purdue University;
F. C. Fenton, Kansas State University;
A. L. Alexander and H. W. Mundt, Naval Research Laboratories;
I. V. Williams and A. Mendizza, Bell Telephone Co.;
W. B. Sinnickson and E. P. Pitman, Port of New York Authority;
R. B. Mears and O. E. Romig, United States Steel Corp.;
A. W. Clyde, The Pennsylvania State College;
R. Price, U. S. Department of Agriculture.

REPORT OF JOINT COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

Meetings:

Meetings of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals, its panels and subcommittees, were held during the past year in New York, N. Y., in conjunction with the Annual Meetings of the American Society for Testing Materials during the week of June 23, 1952, and of The American Society of Mechanical Engineers the week of November 30, 1952.

The Data and Publications, the Aviation, and the Low Temperature Panels met at both Annual Meetings. The Chemical and Petroleum and the Test Methods Panels met in June; the Gas Turbine, the General Research, and the Steam Power Panels met in December.

The Test Methods Panel had additional meetings in Ann Arbor (University of Michigan) in May, 1952, and at the ASTM Spring Meetings in Detroit, Mich., on March 2, 1953. Project 29 had an additional meeting at Columbus (Batelle Memorial Inst.) in September, 1952.

Technical Sessions and Papers:

The following papers were presented at the Annual Meeting of the ASME during the week of November 30, 1952, under the co-sponsorship of the Research Committee on High-Temperature Steam Generation, Power Division, and the Joint Committee on the Effect of Temperature on the Properties of Metals:

Laboratory Investigation of Superheater Tubing Materials in Contact with Synthetic Combustion Atmospheres at 1350 F—C. J. Slunder, A. M. Hall, and J. H. Jackson of Battelle Memorial Institute.

Resistance of Cast Fe-Cr-Ni Alloys to Corrosion in Oxidizing and Reducing Flue-Gas Atmospheres—J. H. Jackson, C. J. Slunder, and O. E. Harder of Battelle Memorial Institute, and J. T. Gow of Electric Steel Foundry Co.

Corrosion of Mercury Boiler Tubes During Combustion of Heavy Residual Oil—A. M. Hall and J. H. Jackson of Battelle Memorial Institute, and D. Douglass of The Hartford Electric Light Co.

Thermal Shock and Other Comparison Tests of Austenitic and Ferritic Steels for Main Steam Piping—W. C. Stewart and W. G. Schreitz, U. S. N. Engineering Experimental Station.

During this Annual Meeting of the ASTM in Atlantic City, a Symposium on "Metallic Materials at Low Temperatures" will be presented by the Low Temperature Panel. The Symposium covers five sessions at which more than 25 papers, covering various aspects of testing and service at low temperatures, are being presented.

It is planned to have these papers produced as a special publication.[1] The committee has adopted the policy of furnishing copies of all papers and publications sponsored by it to all contributors to its financial support.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] To be issued as separate technical publication *ASTM STP No. 158.*

Membership:

By action of the Sponsor Societies, on recommendation of the Committee, Addison M. Rothrock, Assistant Director of Research of the National Advisory Committee on Aeronautics, George V. Smith, United States Steel Corp. Research Laboratory, and Paul M. Brister, Babcock and Wilcox Co., have been appointed to membership on the committee. R. F. Miller of the United States Steel Corp., who has been unusually active in the committee's work as chairman of the Data and Publications Panel, has submitted his resignation from the committee because of pressure of other work. The committee regrets this loss and wishes to record its sincere appreciation of the splendid work he has performed in its behalf for a number of years.

Finances:

During the past year, the committee's campaign for funds under the chairmanship of N. L. Mochel has brought in about $16,000, $10,000 of which was contributed by the Edison Electric Institute. The total now stands at nearly $81,000.

Contracts:

As has previously been reported, four contracts have been entered into for a sum totalling $21,600. Additional contracts are in the making, funds having been authorized as follows:

Project SP2:	"Properties of Cast Iron at Elevated Temperatures."	$10,000
DP5:	"Survey of Relaxation Data"	$1 ,000
DP7:	"Survey of Data on Copper-Base Alloys"	$1 ,000

Total contractural and projected contractural obligations thus total $33,600. The present rate of effort on work in progress appears to call for expenditures of the order of $10,000 per year. Funds in the Custodian Account, administered by the ASME, amounted to $78,986.66 as of May 1, 1953.

ACTIVITIES OF PANELS AND PROJECT SUBCOMMITTEES

Aviation Panel (Leo Schapiro, chairman):

The first report of progress from the University of Michigan on Project AP1, devoted to the creep-rupture testing of 0.040-in. sheets of Types 321 and 347 stainless steel and of N-155 and Inconel X, has been received and is devoted principally to a discussion of means of measuring the large gage length elongations involved. It is expected that work on this project will be completed during the present summer.

The report under project AP2, covering short-time tension and creep-rupture phases of the work, was presented as a paper by D. Preston during the Symposium on Strength and Ductility of Metals at Elevated Temperatures with Particular Reference to Effects of Notches and Metallurgical Changes at the June, 1952, Annual Meeting of the ASTM (issued as separate publication, *ASTM STP No. 128*). The Thermal Shock phase of the work, involving a large amount of data, has also been reported to the Panel.

Data and Publications Panel (George V. Smith, chairman):

Data have been collected for the high-temperature properties survey, Projects DP3 (superalloys) and DP4 (chromium-molybdenum steels). The compilation of the data for the chromium-molybdenum survey has been completed and is now ready for reproduction as a Special Technical Publication. Analysis of the "Superalloy" data has not yet been started.

The gathering of data under Projects DP5 (relaxation), DP6 (strength of weldments at elevated temperatures), and DP7 (copper-base alloys) is under way.

Gas Turbine Panel (*C. T. Evans, chairman*):

Stress-rupture and tension-fatigue tests on notched specimens of N-155 alloy (Project GTP-1) have been practically completed and a progress report has been received from Battelle Memorial Inst. Results indicate that some notch sensitivity may develop in stress rupture tests of N-155 of longer duration than 300 hr at 1200 F but none was indicated in tests at 1350 and 1500 F.

A symposium on the Effect of Cyclic Heating and Cyclic Stressing on Metals at High Temperatures is planned for the 1954 ASTM meeting.

A bibliography on oil ash corrosion and related subjects has been compiled by Chairman Evans and distributed to the Panel members.

The Technical Advisory Committee approved the addition of W. D. Manly, Oak Ridge National Laboratory; J. A. Cameron, Elliott Co.; E. B. Delgass, Socony Vacuum Laboratories; and G. V. Smith, U. S. Steel Corp. Research Laboratory, to membership of the Panel.

General Research Panel (*A. J. Herzig, chairman*):

The Panel is directing its attention to matters bearing on "Basic Effects of Environment on Strength, Scaling, and Embrittlement of Metals at Elevated Temperatures" and may develop a symposium on the subject. It will participate in the symposium planned for 1954 by the Gas Turbine Panel.

Petroleum and Chemical Panel (*C. L. Clark, chairman*):

Project 8, under R. F. Miller, has submitted a preliminary report on "Effect of Manufacturing Practice on High-Temperature Strength of Carbon-Steel Plate and Pipe" indicating that grain-size variations due to melting practice have little effect on rupture strength at 1000 F, but that coarse-grain melting practice produced steel having superior creep resistance at 1000 F.

A new project is under way on "High Temperature Strength of Welded Joints" under the Chairmanship of W. B. Hoyt.

E. C. Miller, Oak Ridge National Laboratory, has been appointed to membership in the Panel.

Steam Power Panel (*P. M. Brister, chairman*):

Project SP2 has been organized and bids have been invited for work on fundamental information on the "Properties of Cast Iron at Elevated Temperatures." Chairman of the project is J. S. Vanick. This work is to comprise a compilation and review of available information and the testing of irons to determine the relative hardness, stress-rupture, creep, and thermal-shock resistance at temperatures up to the A_3 of selected cast irons from Specifications A 278. The tests are expected to furnish data useful in estimating safe loads for elevated temperature service.

Work under SP3 on the problems associated with Type 347 welds in main steam service is to be correlated with similar work being carried on by the Welding Research Council under sponsorship of AEC in order to satisfy the needs of the power industry and yet avoid duplication of effort.

The panel is actively interested in the use of dissimilar metal joints in main steam line service and plans to have two or more papers on the subject for the December, 1953, Annual ASME Meeting.

In view of the adequacy of the high temperature properties of present chromium-molybdenum steels in use in power plant service and the availability of molybdenum under use limitations of

NPA, the study of possible molybdenum reduction in this steel has been abandoned.

E. C. Miller, Oak Ridge National Laboratory, and G. V. Smith, U. S. Steel Corp. Research Laboratory, have been appointed to membership on the Panel.

N. L. Mochel, while retaining panel membership, resigned as its chairman and was succeeded by Paul M. Brister, Babcock and Wilcox Co.

Low Temperature Panel (*Irving Roberts, chairman*):

This panel organized the comprehensive symposium covering five sessions and more than 25 papers on the subject "Metallic Materials at Low Temperatures," as a feature of the current annual meeting.[1]

Test Methods Panel (*J. J. Kanter, chairman*):

The panel has been active for some time on revisions of Recommended Practices for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 - 43), Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 - 41), and Conducting Time-for-Rupture Tension Tests of Metallic Materials (E 85 - 50 T).

Effect of Certain Elements on Graphitization of Steel—Project 29 (*J. J. Kanter, chairman*):

This Project has been completed with a Summary Report from Battelle Memorial Inst. The work covered twelve laboratory heats of steel made up of three base type 0.20 per cent carbon steels—one a pure iron-carbon steel, one a pure iron-carbon steel to which 0.25 per cent chromium had been added, and the other a pure iron-carbon steel to which 0.6 per cent manganese, 0.20 per cent silicon, 0.02 per cent phosphorus, and 0.03 per cent sulfur were added. To one ingot of each of the three types was added nitrogen, to another aluminum, and to another both aluminum and nitrogen—the fourth ingot in each series had no addition made to it. Specimens were rolled and then welded preparatory to exposure at 1025 F for periods of up to 8000 hr to induce graphitization of the carbon. Aluminum, as expected, promoted graphitization, nitrogen increased resistance to graphitization at 1025 F. Whether or not the aluminum present in the metal exceeded the nitrogen content appeared to be a factor. Strangely enough, the 0.25 per cent chromium steel graphitized more readily than the pure carbon-iron steel.

Respectfully submitted on behalf of the committee,

F. B. FOLEY,
Chairman.

HOWARD C. CROSS,
Secretary.

V. T. MALCOLM,
Vice-Chairman.

REPORT OF AWS-ASTM JOINT COMMITTEE

ON

FILLER METAL*

The Joint AWS-ASTM Committee on Filler Metal has continued active work on its assignment to prepare specifications for both ferrous and non-ferrous arc welding electrodes and gas welding rods. A summary of the activities of the several subcommittees follows:

Subcommittee I on Iron and Steel Filler Metal (E. H. Turnock, chairman).—Tentative Specification ASTM A 233 – 48 T and AWS A 5.1 – 48 T for mild steel arc welding electrodes has been considered as still adequately serving its purpose, so no work of revision has been done. The same is true of Tentative Specification ASTM A 251 – 46 T and AWS A 5.2 – 46 T for iron and steel gas welding rods. Both of the above specifications will be brought up for reconsideration in the coming year.

The working group of Subcommittee I has held several meetings during the past year in preparation of a revised specification covering low-alloy steel arc welding electrodes. A few points remain to be cleared in a meeting scheduled for April 22, 1953, prior to submission to the full committee and then to the main joint committee. The work should be completed and the specification published this year.

Subcommittee III on Aluminum and Aluminum Alloy Filler Metal (G. O. Hoglund, chairman) has held several meetings during the year and is nearing completion of its work in preparing tentative specification for aluminum and aluminum-alloy welding rods and bare electrodes. A meeting is scheduled for April 17, 1953, to clear the last few points before sending it to the main joint committee for approval. Publication is expected this year.

Subcommittee IV on High Alloy Steel Filler Metal (R. D. Thomas, Jr., chairman) has held several meetings during the past year on preparing a specification on bare corrosion-resisting chromium and chromium-nickel steel welding rods and electrodes. Submission for main joint committee approval is anticipated soon. It should be published this year.

The joint specification on corrosion-resisting chromium and chromium-nickel steel welding electrodes, ASTM A 298 – 48 T and AWS A 5.4 – 48 T, will be brought up for review and modification this year.

Subcommittee V on Nickel and Nickel-Alloy Filler Metal (O. B. J. Fraser, chairman) held one meeting during the year, following which a revised specification draft has been prepared for early submission to the subcommittee.

Subcommittee VI on Copper and Copper-Alloy Filler Metal (F. E. Garriott, chairman).—A newly revised specification for copper and copper-alloy welding electrodes, to supersede ASTM B 225 – 48 T and AWS A 5.6 – 48 T, is in its final stages before being submitted to the joint main committee. Publication is expected this year.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

Subcommittee VII on Surfacing Materials (H. S. Avery, chairman) has continued work on the preparation of a tentative specification for electrodes for surfacing metals and a tentative specification for welding rods for surfacing metals.

Each of the proposed specifications divides the materials into several types on the basis of chemical composition, with appendices for these various types dealing with the properties of the materials and the precautions necessary in their application.

Subcommittee VIII on Brazing Filler Metal (A. N. Kugler, chairman) completed work on the Tentative Specification for Brazing Filler Metal, which has been approved by the ASTM Administrative Committee on Standards and the Board of Directors of AWS. The designations ASTM B 260 - 52 T and AWS A 5.8 - 52 T have been assigned to the Specification. The committee's work this coming year will consist of following its acceptance by industry and noting possible changes to be considered in the future.

This report has been submitted to letter ballot of the committee, which consists of 64 members; 47 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. H. TURNOCK,
Chairman.

S. A. GREENBERG,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the Joint Committee on Filler Metal presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specification for:

Corrosion-Resisting Chromium and Chromium-Nickel Steel Welding Rods and Bare Electrodes (A 371 - 53 T).

Revision of Tentative Specification for:

Copper and Copper-Alloy Metal Arc-Welding Electrodes (B 225 - 48 T).

Withdrawal of Standard Specification for:

Silver Solders (B 73 - 29).

The recommendation with respect to the withdrawal of Standard Specification B 73 was accepted by the Standards Committee on September 9, 1953; the Tentative Specification A 371 and revision of Tentative Specification B 225 were accepted on December 16, 1953. The new Specification A 371 appears in the 1953 Supplement to Book of ASTM Standards, Part 1, and the revised Specification B 225 is available as a separate.

REPORT OF COMMITTEE C-1

ON

CEMENT*

Committee C-1 on Cement held three meetings during the past year: in New York, N. Y., June 26, 1952; at Philadelphia, Pa., October 31; and in Detroit, Mich., March 4, 1953.

The Philadelphia meeting was held just 50 years after the first meeting of the committee, which also took place in Philadelphia. On October 30 there was a well-attended Anniversary Dinner, with appropriate speeches supplemented by the remarks of Honorary Members of the committee. A souvenir booklet commemorating the occasion offered a brief review of the history of Committee C-1, and contained individual pictures of all of the current members of the committee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-1 presented to the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 51 T).

Tentative Revision of Standard Specifications for:

Masonry Cement (C 91 – 51).

These recommendations were accepted by the Standards Committee under its actions of September 5, 1952, and appear in the 1952 Book of ASTM Standards, Part 3.

NEW TENTATIVE

The committee recommends the acceptance of the proposed Tentative Method for Mechanical Mixing of Hydraulic-Cement Mortars of Plastic Consistency as appended hereto.[1] After long interest in and extensive studies of mechanical mixing of standard test mortars, this method is now being promulgated. The specified mixer has been used in numerous cooperative tests, and the procedure is based on various closely similar methods employed in those studies in recent years. The recommended tentative is essentially the same as the method under consideration as a Federal specification.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends revisions for immediate adoption in the following standards, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Portland Cement (**C 150 – 52**).[2]—In Table II, under Air Content of Mortar, change the per cent value "15" to read "12" for all five types of cement. This change has appeared very desirable in order to provide an appropriate difference between the lower limit for entrained air under Tentative Specifications for Air-Entraining Portland Cement (C 175 – 52 T) and the upper limit under Specification C 150. The recommended change also would effect agreement with the Federal specifications on this point.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

[2] 1952 Book of ASTM Standards, Part 3.

Standard Specifications for Masonry Cement (**C 91 – 51**).[2]—Four changes are recommended for immediate adoption in order to bring the procedure for the determination of strength more closely in accord with the corresponding procedure in the Standard Test for Compressive Strength of Hydraulic-Cement Mortars (ASTM Designation C 109). The revisions are:

Section 19 on Apparatus.—Add the following new paragraph:

> "(*h*) *Tamper.*—The tamper shall be made of a nonabsorptive, nonabrasive material such as medium-hard rubber or seasoned oak wood rendered nonabsorptive by immersion for 15 min in paraffin at approximately 200 C (392 F) and shall have a cross-section of ½ by 1 in., and a convenient length (5 or 6 in.). The tamping face of the tamper shall be flat and at right angles to the length of the tamper."

Reletter the present Paragraphs (*h*) and (*i*) to read (*i*) and (*j*) respectively.

Section 24.—Delete the present wording and replace with: "The flow shall be determined in accordance with Section 9 of Standard Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 52)."

Section 25.—Delete the present wording and replace with: "Molding of test specimens shall be in accordance with Section 10 of Standard Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 52)."

Section 27.—Delete the present wording and replace with new Paragraphs (*a*)(*b*)(*c*) that will read exactly the same as in Section 12 of Standard Method C 109, except that the first line of Paragraph (*a*) shall read: "Testing of the cube specimens will be carried out immediately after their removal from the moist closet for 7-day specimens, and from storage water for all other specimens." This change in Section 27 relates to the testing of the compressive strength specimens, and would make the rate of loading requirements the same as under Standard Method C 109.

REVISION OF TENTATIVES

Tentative Method of Test for Bleeding of Cement Pastes and Mortars (**C 243 – 52 T**).[2]—The committee recommends the acceptance of the revisions given below. The changes are designed to effect certain desirable refinements in details of procedure and provide for the important change from hand-mixing to machine mixing of the pastes and mortars. The revisions are as follows:

Section 4(a).—In the first part of the second sentence, change the present batch weights of "1000 g of cement and 3000 g of natural silica sand" to 750 g and 1875 g respectively. In the latter part of the same sentence, relating to the method of determining the amount of mixing water for the mortar, change the present flow limits, 100 and 115, to read 105 and 110, respectively.

Section 4(b).—Delete the present first sentence that describes hand-mixing and substitute therefor the following three new sentences and footnote:

> "The mixing shall be done in an epicyclic type mechanical mixer having a positive mechanical speed control which can be set to give a speed of between 135–150 rpm.[a] The mixing bowl capacity shall be not less than 3 qt and not more than 5 qt. A flat type beater designed to conform to the mixing bowl shall be used.
>
> ---
>
> [a] A mixer such as the Hobart C-10 with proper adapters, or the Hobart N-50 is considered satisfactory."

Appropriate editorial changes will need to be made elsewhere in the text where required by the foregoing changes.

Section 6.—Add the following new initial paragraph: "(*a*) *Vibration.*—The bleeding apparatus shall be placed in a location and set on a sponge rubber mat or equivalent which will serve as a vibration damper."

Designate the present Section 6 as Paragraph "(*b*) *Ventilation.*"

Tentative Method of Test for Air-Content of Hydraulic Cement Mortar (**C 185 - 50 T**).[2]—The committee recommends the acceptance of the revision of this tentative method, as appended hereto.[3] The principal revisions are the substitution of mechanical mixing for the earlier hand-mixing of the mortar, and the substitution of the flow table for the Burmister flow trough. It is felt that the mechanical mixing of the standard mortar will effect greater reproducibility in results than was possible with the hand-mixing. The adoption of the flow table in place of the Burmister trough as the means of determining the amount of mixing water for the mortar was based chiefly on the greater capability of the flow table for adjustment and calibration, and also on the fact that this change would result in only one piece of equipment for determining the consistency of the standard mortars. The flow table is also used in the air-entrainment test method in the Federal methods of testing cement. This revision of Tentative Method C 185 was the outgrowth of an extensive program of tests, in which twelve laboratories participated, using seven cements, and studying numerous details of procedures and apparatus.

Tentative Specification for Portland-Blast Furnace Slag Cement (**C 205 - 51 T**).[2]—The committee recommends the acceptance of the following revisions. The adoption of these changes will make the setting time requirements of this specification consistent with those of Standard Specification for Portland Cement (C 150) and Tentative Specifications for Air-Entraining Portland Cement (C 175 T), and would make use of the revised Vicat test method.

Table II, Section 5.—Revise the setting time requirements to read as follows:

Time of Setting	Type IS	Type IS-A
Vicat test (Method C 191)		
Set, min, not less than..	45	45
Set, hr, not more than...	7	7

Section 11(f).—Revise to read "*Time of Setting.*—Standard Method of Test for Time of Setting of Hydraulic Cement by Vicat Needle (ASTM Designation: C 191)."

TENTATIVE REVISION OF STANDARDS

Standard Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (**C 204 - 51**)[2].—The committee recommends the acceptance of the following tentative revisions in the interest of improvement and clarification:

Section 3(b), Paragraph 2.—Delete "pencil or thin" and insert "slightly smaller than the cell diameter" after "rod" in the first sentence.

Paragraph 3.—Change "0.01" to "0.005."

Table I.—Insert "Note 3" after "Mercury" in the title and in the heading of Column 2 after "Mercury." Add the following new note just after present Note 2.

"NOTE 3.—The density of the mercury shall be determined."

Paragraph 4.—Change "0.01" to "0.005."

Section 3(e), line 8.—Change "0.01 g" to "0.001 g."

Section 3(f), Paragraph (1).—Change "Note 3" to "Note 4."

Paragraph (3).—Change "Note 4" to "Note 5;" "Note 3" to "Note 4;" and "Note 4" to "Note 5."

Section 3(g).—Delete the entire section and designate the present Section 3(*h*) as Section 3(*g*).

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[3] The revised tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

Section 5.—Delete the section and replace with the following:

"Calculation of specific surface values shall be made according to the following formulas":

$$S = \frac{S_S\sqrt{T}}{\sqrt{T_S}} \tag{3}$$

$$S = \frac{S_S\sqrt{n_S}\,\sqrt{T}}{\sqrt{T_S}\,\sqrt{n}} \tag{4}$$

$$S = \frac{S_S(1-e)_S\sqrt{e^3}\,\sqrt{T}}{\sqrt{e^3_S}\,\sqrt{T_S}\,(1-e)} \tag{5}$$

$$S = \frac{S_S(1-e)_S\sqrt{e^3}\,\sqrt{n_S}\,\sqrt{T}}{\sqrt{e^3_S}\,\sqrt{T_S}\,\sqrt{n}\,(1-e)} \tag{6}$$

$$S = \frac{S_S\,p_S(1-e)_S\sqrt{e^3}\,\sqrt{T}}{p(1-e)\sqrt{e^3_S}\,\sqrt{T_S}} \tag{7}$$

$$S = \frac{S_S\,p_S(1-e)_S\sqrt{n_S}\,\sqrt{e^3}\,\sqrt{T}}{p(1-e)\sqrt{e^3_S}\,\sqrt{T_S}\,\sqrt{n}} \tag{8}$$

where:

S = the specific surface in sq cm per g of the test sample,

S_S = the specific surface in sq cm per g of the standard sample used in calibration of the apparatus,

T = measured time interval, in seconds, of manometer drop for test sample (Note),

T_S = measured time interval in seconds of manometer drop for standard sample used in calibration of the apparatus (Note),

n = viscosity of air in poises at the temperature of test of the test sample (Note),

n_S = viscosity of air in poises at the temperature of test of the standard sample used in calibration of the apparatus (Note),

e = porosity of prepared bed of test sample (Note),

e_S = porosity of prepared bed of standard sample used in calibration of apparatus (Note),

p = specific gravity of test sample (for portland cement a value of 3.15 shall be used), and

p_s = specific gravity of standard sample used in calibration of apparatus (assumed to be 3.15).

NOTE.—Values for $\sqrt{n}$, $\sqrt{e^3}$ and $\sqrt{T}$ may be taken from Tables I, II, and III respectively.

Equations 3 and 4 shall be used in calculations of fineness of portland cements compacted to the same porosity as the standard fineness sample. Equation 3 is used if the temperature of test of the test sample is within ±3 C of the temperature of calibration test of the standard fineness sample, and Eq 4 is used if the temperature of tests is outside of this range.

Equations 5 and 6 shall be used in calculation of fineness of portland cements compacted to some porosity other than that of the standard fineness sample used in the calibration test. Equation 5 is used if the temperature of test of the test sample is within ±3 C of the temperature of calibration test of the standard fineness sample, and Eq 6 is used if the temperature of tests is outside of this range.

Equations 7 and 8 shall be used in calculation of fineness of materials other than portland cement. Equation 7 shall be used when the temperature of test of the test sample is within ±3 C of the temperature of calibration test of the standard fineness sample, and Eq 8 is used if the temperature of tests is outside of this range.

Standard Specification for Masonry Cement (**C 91 – 51**)[2].—The committee recommends the acceptance of the following tentative revision in order to provide a more stringent soundness test requirement for masonry cement. The recommendation is made after a considerable period of tests and consideration.

Section 3.—Delete and replace with the following new section:

"Masonry cement of each of the two types shown under Section 4 shall not expand more than 1 per cent when tested as prescribed in Section 18."

Section 18.—Delete and replace with the following new section:

"Soundness shall be determined by the Standard Method of Test for Autoclave Expansion of Portland Cement (ASTM Designation: C 151), except that after molding the bars shall be stored in moist cabinet for 48 hr ± 30 min; then an initial length measurement shall be made and the bars be returned to moist cabinet storage for an additional 5 days, at the end of which time the bars shall be autoclaved. The difference between the length of the test specimen at 48 hr and the length after autoclaving at the age of 7 days shall be calculated to the nearest 0.01 per cent of the effective gage length, and shall be reported as the autoclave expansion of the cement."

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the following tentative revisions of standards be adopted as standard:[2]

Standard Specification for Masonry Cement (**C 91 – 51**). This revision relates to packaging and marking and was mentioned earlier in this report as having been accepted by the Administrative Committee on Standards.

Standard Method of Test for Portland Cement by the Turbidimeter (**C 115 – 42**). This tentative revision was issued June, 1951.

Standard Method of Test for Heat of Hydration of Portland Cement (**C 186 – 49**). This tentative revision was issued March, 1950.

Standard Method of Test for Autoclave Expansion of Portland Cement (**C 151 – 49**). The tentative revision was issued September, 1951.

Standard Methods of Chemical Analysis of Portland Cement (**C 114 – 47**). This tentative revision was issued September, 1951.

Standard Specification for Portland Cement (**C 150 – 52**). These tentative revisions were related to the SO_3 content and the fineness, and were issued September, 1951 and January, 1952 respectively.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends the adoption as standard of the Tentative Specification for Air-Entraining Portland Cement (**C 175 – 52T**),[2] which was issued in 1942 and has been revised several times since then.

The committee also recommends the adoption as standard of the definition of the term "Addition," which forms part of Tentative Definitions of Terms Relating to Hydraulic Cement (**C 219 – 51 T**).[2] The remainder of this tentative is being recommended elsewhere in this report for retention as tentative.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives[2] be continued as tentative without revision, except for certain noted editorial revisions:

Tentative Methods of Test for:

Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 – 51 T),
Time of Setting of Hydraulic Cement by Gillmore Needles (C 266 – 51 T),
Time of Setting of Hydraulic Cement in Mortar (C 229 – 51 T), and
Sodium Oxide and Potassium Oxide in Portland Cement by Flame Photometry (C 228 – 49 T).

Tentative Methods of Chemical Analysis of:

Portland Cement (C 114 – 51 T), consisting of the methods for Vinsol Resin, Darex, and Titanium Oxide.

Definition of the term "Pozzolan," subject to editorial change of the words "siliceous or aluminous" to read "siliceous or siliceous and aluminous." This definition forms part of Tentative Definitions of Terms Relating to Hydraulic Cements (**C 219 – 51T**)[2]. The other part of that tentative is being recommended for advancement to standard elsewhere in this report.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Sponsoring Committee on Portland Cement (C. H. Scholer, chairman) has been studying specification questions, particularly those relating to the limits for air-entrainment. The work has resulted in a number of recommendations affecting standards and now presented in this report.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Sponsoring Committee on Masonry Cement (E. J. Wechter, chairman) has reviewed details of specifications and has presented recommendations affecting standards, particularly the tentative revision of specification C 91, providing an autoclave test requirement for masonry cement.

Sponsoring Committee on Blended Cement (L. R. Forbrich, chairman) has actively continued its study of specifications for fly ash and for portland-pozzolan cements. Recommendations were made affecting tentative specification C 205.

Editorial Committee (W. J. McCoy, chairman) has continued its review of proposed new standards and revisions in standards and also handled the preparation of the Fiftieth Anniversary booklet.

Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) is considering questions relating to flame photometry and is continuing its search for a better method for manganese removal in the analysis of cement.

Working Committee on Volume Change and Soundness (Herbert Insley, chairman) has studied details relating to the methods under its supervision, and has made recommendations affecting Method C 151.

Working Committee on Sulfate Resistance (William Lerch, chairman) has continued its study of the significance of the results of the recent extensive series of cooperative tests sponsored by the subcommittee.

Working Committee on Time of Setting (E. E. Berger, chairman) has been engaged in the study of questions relating to the test methods within the scope of activity of this subcommittee.

Working Committee on Heat of Hydration (W. J. McCoy, chairman) is engaged in the further study of a rapid, simplified procedure that is similar to the method in the recent Federal Specifications SS-C-158c.

Working Committee on Bleeding, Plasticity and Workability (H. L. Kennedy, chairman) has studied variables involved in Method C 243 and has offered recommendations affecting that method, including the substitution of mechanical mixing for hand mixing of the mortars.

Working Committee on Fineness (R. L. Blaine, chairman) has offered revisions affecting Method C 204 and has continued its study of methods for determining fineness and density of finely powdered materials.

Working Committee on Strength (C. E. Wuerpel, chairman) has reviewed the recently proposed tests for flexural strength of mortars and for the compressive strength of mortars using portions of the prisms broken in flexure. The subcommittee has also outlined a series of cooperative tests to determine the effect of substituting mechanical mixing in place of hand mixing in preparing the mortars for these two tests and also for the standard (C 109) test for compressive strength of mortar cubes. It is expected that nine laboratories will participate in the proposed series of tests, using about 25 cements.

Working Committee on Additions (H. W. Russell, chairman) has contributed a number of proposed revisions in Specification C 226, which recommendations were accepted by the Administrative Committee on Standards.

Working Committee on Coordination of Methods of Test (J. R. Dwyer, chairman) reviewed the methods and apparatus used and the data secured in some of the cooperative mortar tests in which mechanical mixing had been included. As a result of the study, the subcommittee assembled a recommended uniform specification for the apparatus and method to be used in mixing standard mortars. It is hoped that the mechanical mixing will greatly aid in securing more uniform results in the various tests of standard mortars, and the proposed method is appended to this report as a recommended new tentative.[1]

Working Committee on SO_3 Content (H. S. Meissner, chairman) has been considering the experience thus far in the use of Method C 265 - 51 T as a test for calcium sulfate in hydrated portland-cement mortar. Certain features of the method are to be studied by the subcommittee in a cooperative series of tests.

Working Committee on Methods of Test for Air-Entrainment (H. K. Cook, chairman) issued a report of the results obtained in the extensive cooperative test program in which 12 laboratories had used 7 cements in studying numerous details of apparatus and methods employed in the determination of entrained air in standard mortar. As a result of those studies, the subcommittee offered the revision of Method C 185, as appended hereto.[3]

Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) reported that the laboratory is continuing its regular inspection service among cement laboratories, and has arranged for the early distribution of a comparative test sample to an estimated 200 interested laboratories The laboratory has collaborated with various other subcommittees in matters related to apparatus and methods and has begun the necessary editing for the next edition of "ASTM Standards on Cement." Numerous samples of the flow table calibration material had been distributed.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members, of whom 74 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. S. WEAVER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard:

Specifications for Masonry Cement (C 91 - 51), and
Method of Test for Normal Consistency of Hydraulic Cement (C 187 - 49).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the tentative revisions appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-2

ON

MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride Cements met twice during the year: on June 26, 1952 in New York, N. Y., and on May 5, 1953, in Washington, D. C.

Of special interest to the committee is the action taken at the last meeting of Sectional Committee A88 on Magnesium Oxychloride Cement Flooring, of the American Standards Association. This Committee recommended that all ASTM Standards on Magnesium Oxychloride and Magnesium Oxysulfate Cements be submitted to ASA for consideration and approval as American Standards.

ADOPTION OF TENTATIVES AS STANDARD

Committee C-2 recommends that the following two tentatives be adopted as standard by the Society without revision:

Tentative Specifications for:

Magnesium Chloride (C 276 – 51 T),[1] and
Magnesium Sulfate (C 277 – 51 T).[1]

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the following tentative be continued without revision:

Tentative Specification for:

Oxychloride Magnesia (C 275 – 51 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot vote of the committee, which consists of 20 members; 17 members returned ballots, of whom 15 have voted affirmatively and 0 negatively.

Respectively submitted on behalf of the committee,

L. S. WELLS,
Chairman.

J. B. JAMES,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-3

ON

CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held three meetings during the year: in New York, N. Y., June 25, 1952, in connection with the 1952 Annual Meeting of the Society; at ASTM Headquarters, Philadelphia, Pa., October 29, 1952; and in New York, N. Y., March 11, 1953.

The membership of Committee C-3 now consists of 23 members, of whom 10 are classified as producers, 6 as consumers, and 7 as general interest members. There are 4 consulting members.

NEW TENTATIVES

The committee recommends that the following three methods be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:

Compressive Strength of Resin Type Chemical-Resistant Mortars,
Tensile Strength of Resin Type Chemical-Resistant Mortars, and
Working and Setting Times of Resin Type Chemical-Resistant Mortars.

CONTINUATION OF TENTATIVE WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Chemical Resistance of Hydraulic-Cement Mortars (C 267 - 51 T) be retained as tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VIII on Bond Strength (J. R. Allen, chairman) reports that the development of a new test method for bond strength has been completed and will be submitted to the committee for consideration during the ensuing year.

Subcommittee IX on Chemical Resistance (R. H. Steiner, chairman).—A test method for chemical resistance has been formulated and submitted to the committee for consideration. Several organizations are making duplicate tests to confirm the results obtained to date with this procedure.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BEAUMONT THOMAS,
Chairman.

J. R. ALLEN,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE*

Committee C-4 held one meeting during the year on June 24, 1952, at the time of the Annual Meeting of the Society.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards proposed Tentative Methods of Testing Clay Pipe. The methods were accepted by the Standards Committee on September 5, 1952, and appear in the 1952 Book of ASTM Standards, Part 3, bearing the designation C 301 – 52 T.

The description of the test procedures appearing in the clay pipe specifications (C 13 – 50 T, C 200 – 50 T, C 211 – 50, C 261 – 50 T and C 278 – 51 T) were deleted editorially in 1952 and replaced by references to the new tentative methods.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Specifications (J. O. Everhart, chairman) was instructed to give further study to certain provisions of the test procedures shown in Tentative Methods of Testing Clay Pipe (C 301 – 52 T).

Subcommittee III on Clay Flue Lining (Wendell Anderson, chairman) has made some progress in the dimensional field as the first step toward developing a draft of standards for clay flue linings. The subcommittee was instructed to prepare a recommended list of standard sizes of flues to be published in the ASTM BULLETIN for information purposes only and to solicit recommendations.

This report has been submitted to letter ballot of the Committee, which consists of 30 members; 26 members returned their ballots, of whom 25 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JOHN C. RIEDEL,
Chairman.

ROBERT E. SCOTT,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specification for:

Clay Flue Lining (C 315 – 53 T).

Revision of Tentative Specification for:

Extra Strength Clay Pipe (C 200 – 50 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the past year: the first at New York, N. Y., on June, 1952, during the 1952 Annual Meeting, and the second in Detroit, Mich., on March 6, 1953, during ASTM Committee Week. The committee now consists of 22 producers, 19 general interest members, and 6 consumers.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of:

Standard Specification for Sand for Use in Plaster (C 35 – 39), and
Standard Specification for Quicklime (C 53 – 39).

These recommendations were accepted by the Standards Committee on September 5, 1952, and December 12, 1952, respectively, and appear in the 1952 Book of ASTM Standards, Part 3. The revision of Specification C 35 – 39 was prepared jointly with Committee C-11.

TENTATIVE REVISION OF STANDARD

The committee recommends as a tentative revision of the Standard Methods of Physical Testing of Quicklime and Hydrated Lime (**C 110 – 49**)[1] the following new procedure for determining the settling rate of hydrated lime to be added, when adopted, at the end of the methods as Section 18:

18. Settling Rate of Hydrated Lime: (*a*) *Procedure.*—Place 10.0 g of lime hydrate in a 100-ml glass-stoppered graduate cylinder (internal diameter about 2.4 cm). Wet with 50 ml of CO_2-free distilled water at 73 ± 3 F and mix thoroughly by alternately inverting and righting the cylinder slowly for a period of 2 min. Allow the graduate and contents to stand at 73 ± 3 F for 30 min and then make up to the 100-ml mark with CO_2-free distilled water at 73 ± 3 F. Mix contents again thoroughly as before and allow to stand undisturbed at 73 F ± 3 F for 24 hr.

(*b*) *Report.*—Report the sedimentation height in milliliters after $\frac{1}{4}$, $\frac{1}{2}$, $\frac{3}{4}$, 1, 2, 4, and 24 hr, reading the bottom of the meniscus.

NOTE.--Slight variations in results of this test on a sample run in different laboratories or by different operators are permissible. The test is not an absolute one, but is designed to distinguish between fast and slow settling hydrates.

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

The subcommittees are developing a new specification for lime for soil stabilization, undertaking round-robin tests on various autoclave tests for soundness of limes, and checking new available lime, iron, and versenate methods of tests for lime.

This report has been submitted to letter ballot of the committee; 35 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JAMES A. MURRAY,
Chairman.

ROBERT S. BOYNTON,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Inorganic Aggregates for Use in Interior Plaster (C 35 – 52 T).*

Modification of Tentative Revision of:

Standard Specifications for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 42).

Withdrawal of Standard Specifications for:

Quicklime and Hydrated Lime for Use in the Textile Industry (C 48 – 24).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the revised tentative and tentative revision appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

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* Jointly with Committee C-11 on Gypsum

REPORT OF COMMITTEE C-8

ON

REFRACTORIES*

Committee C-8 on Refractories has held two meetings during the past year: the eighty-second at French Lick Springs, Ind., on September 12, 1952, and the eighty-third in Detroit, Mich., on March 4, 1953.

The committee has lost by death Hewitt Wilson of the Bureau of Mines; by retirement, B. A. Hollenbeck of Standard Oil Development Co.; and by resignation, J. W. Whittemore of Virginia Polytechnic Inst. New members are A. J. Metzger of Virginia Polytechnic Inst. and the Special Refractories Assn., represented by G. B. Remmey, who was previously a personal producer member.

The present membership is 36, of whom 20 are classified as producers, 10 as consumers, and 6 as general interest members.

TENTATIVE REVISION OF STANDARDS

Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (**C 133 – 49**).[1] —The committee recommends the following tentative revision:

Section 8, Test Specimens.—Add the following sentence: "An original surface, preferably unbranded, shall be used whenever possible for the tension face, that is, the face in contact with the two bottom bearing cylinders."

Definitions of Terms Relating to Refractories (**C 71 – 51**).[1]—The committee recommends the following tentative revisions:

Replace the definition of "Refractories (noun)" with the following: "Materials, usually non-metallic, used to withstand high temperature." Replace the definition of "Refractory (adjective)" with the following: "Resistant to high temperature."

ADOPTION OF TENTATIVE REVISION AS STANDARD

Definitions of Terms Relating to Refractories (**C 71 – 51**)[1].—The committee recommends adoption as standard of the following tentative revision:

Delete the present definition for "Spalling of Refractories," including the Note, and replace it with the tentative revision issued June, 1951.[1]

REAFFIRMATION OF STANDARDS

The committee recommends that the following standards[1] which have stood without revision for six or more years be reaffirmed as standard:

Standard Specifications for:

Ground Fire Clay as a Mortar for Laying-Up Fireclay Brick (C 105 – 47),

Fireclay Plastic Refractories for Boiler and Incinerator Services (C 176 – 47), and

Air-Setting Refractory Mortar (Wet Type) for Boiler and Incinerator Services (C 178 – 47).

Standard Classification for:

Insulating Fire Brick (C 155 – 47).

Standard Method of Test for:

True Specific Gravity of Refractory Materials (C 135 – 47),

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

Workability Index of Fireclay Plastic Refractories (C 181 – 47),
Thermal Conductivity of Insulating Fire Brick (C 182 – 47),
Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (C 198 – 47),
Refractoriness of Air-Setting Refractory Mortar (Wet Type) (C 199 – 47),
Thermal Conductivity of Refractories (C 201 – 47), and
Thermal Conductivity of Fireclay Refractories (C 202 – 47).

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives[1] which have stood without revision for two or more years be retained as tentative:

Tentative Methods of Test for:

Modulus of Rupture of Castable Refractories (C 268 – 51 T), and
Permanent Linear Change on Firing of Castable Refractories (C 269 – 51 T).

EDITORIAL CHANGES

The committee recommends that the following three methods be editorially revised as indicated:

Methods of Chemical Analysis for Refractory Materials (**C 18 – 52**),[1] and *Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials* (**C 24 – 46**).[1]— Add to Section 3 of C 18, and to Section 2(*a*) of C 24, the following sentence: "Precautions shall be taken to prevent contamination of the sample by steel particles from the sampling equipment during crushing or grinding."

Tentative Method of Test for Disintegration of Fireclay Refractories in an Atmosphere of Carbon Monoxide (**C 288 – 52 T**):[1]

Section 2(c).—Add the following note:

> NOTE.—As a precaution against the possibility of tank explosions, the tanks should be stored and used outdoors or in a separate building designed to reduce the explosion hazard.

As a matter of record the committee records below editorial changes which have been made in this method.

Section 3(b).—The sentence now reading "The size of the specimens shall be 9 in. long and $2\frac{1}{2}$ or 3 in. in cross-section," shall read "The size of the specimens shall be 9 in. long and $2\frac{1}{2}$ or 3 in. square in cross-section."

Section 5.—Change to read as follows:

> 5. *Report.* Report the condition of each specimen at each inspection by describing the extent of any spotting and indicating the degree of disintegration classified as follows:
>
> (*1*) *Unaffected,* when no particles spall and no cracking occurs,
>
> (*2*) *Slight disintegration,* when spalls are less than $\frac{1}{8}$ in. in diameter and cracking is slight,
>
> (*3*) *Partial disintegration,* when spalls are larger than $\frac{1}{8}$ in. in diameter and cracking is considerable, or
>
> (*4*) *Complete disintegration,* when the specimen is broken into more than four large pieces.

The committee requests that all standards under jurisdiction of Committee C-8 be recommended to the American Standards Association for adoption as American Standard.

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) has made progress with revision of surveys now in the Manual and preparation of new items for the next edition. Nine surveys are now on the agenda.

Subcommittee II on Research (S. M. Swain, chairman) has presented a series of problems for research connected with the testing of refractories. These are being published in the ASTM BULLETIN.[3]

Subcommittee III on Tests (S. M.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] ASTM BULLETIN, No. 191, July, 1953, p. 42; No. 192, September, 1953, p. 24; No. 193, October, 1953, p. 23; No. 194, December, 1953, p. 40.

Phelps, chairman) has approved the recommendations on methods of tests given elsewhere in this report.

Section (A) on Load (L. J. Trostel, chairman) has continued its study of the methods and has recommended one revision. It is looking into the measurement of modulus of rupture of trapezoidal and circle brick.

Section B on Spalling (R. S. Bradley, chairman) is examining the effects of rate of heating on the spalling test, and considering a spalling test for high-fired blast-furnace brick. Search for a mortar for the spalling panel has turned up nothing better than the kaolin now in use.

Section C on Temperature (J. L. Carruthers, chairman) has decided against eliminating the operation of removing iron, introduced by crushing or grinding, by means of a magnet, as had been previously recommended. The Section believes that the present unintended removal of magnetic material such as magnetite, which forms part of the specimen, will have less effect on the P.C.E. test than would be produced by leaving particles of iron or iron alloys in the sample. At the same time a warning is being added editorially, calling attention to the importance of using machinery and methods that will not introduce abnormal amounts of iron.

Section E on Analysis (H. A. Heiligman, chairman) is starting a cooperative study of the determination of alkalies in refractories by the flame photometer method.

Subcommittee IX on Classifications (A. H. Couch, chairman) has begun work on a revision of the Standard Classification of Fireclay Refractories (C 27 – 41), which will give more detailed classifications. The subcommittee completed a proposed Tentative Classification for Single and Double Screened Ground Refractory Materials which has been accepted by the Committee and will be presented to the Society through the Administrative Committee on Standards.

Subcommittee XV on Basic Granular Refractories (J. J. Hazel, chairman) has begun a study of tests for refractory dolomite, as the first of the materials coming under its jurisdiction.

A special subcommittee on defining what is meant by a *Classification* (J. D. Sullivan, chairman) has cooperated with the Administrative Committee on Standards in the development of a definition which has now been approved by the Administrative Committee for use by ASTM Technical Committees.

This report has been submitted to letter ballot of the committee, which consists of 36 members, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

ROBERT B. SOSMAN,
Chairman.

W. R. KERR,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards a proposed Tentative Classification for Single- and Double-Screened Ground Refractory Materials. This recommendation was accepted by the Standards Committee on September 9, 1953, and the new tentative classification appears in the 1953 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 316 – 53 T.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held three meetings last year: on June 27, 1952, during the Annual Meeting of the Society in New York, N. Y.; on October 9, 1952, at Philadelphia, Pa.; and on March 6, 1953, in Detroit, Mich., during Committee Week of the Society.

On July 30, 1952, the committee suffered the loss through death of Harry J. Love, who had been an active member since 1918 and an Honorary Member since 1949.

The Sanford E. Thompson Award, established by the committee in 1938 in honor of its first chairman, is made for a paper of outstanding merit on concrete and concrete aggregates presented before the Society. The award this year will be made to Mrs. Katharine Mather, Waterways Experiment Station, Corps of Engineers, for her paper entitled "Applications of Light Microscopy in Concrete Research," which was presented at the 1952 Annual Meeting.[1]

New Tentatives

The committee recommends that the following be accepted for publication as tentative, as appended hereto:[2]

Tentative Methods of:

Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water, and

Test for Fly Ash as an Admixture for Portland-Cement Concrete.

Tentative Specifications for:

Liquid Membrane-Forming Compounds for Curing Concrete.

Revision of Tentatives

The committee recommends for revision and continuation as tentative the following:

Tentative Method of Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete **(C 156 – 52 T)**:[3]

Section 2.—In Paragraph (*b*), delete Note 2 relative to the use of $MgCl_2$ for control of humidity in curing cabinet.

Section 4.—Terminate the first sentence after "tamped 50 times," thereby eliminating reference to Section 2(*c*) which is to be deleted (as recommended above).

Section 7.—In the fifth line of Paragraph (*a*), after "brush" and before "(Note 1)," insert the following sentence:

> If surface water appears upon brushing, the specimen shall be returned to the cabinet and removed therefrom immediately upon disappearance of the surface water brought to the surface by the brushing operation, and again brushed.

Delete the last sentence of Paragraph (*a*) which reads, "The specimen shall then be returned to the cabinet." This requirement would be incorrect since the specimen at this point is ready to be weighed, covered with the curing material, and then returned to the cabinet.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Published in Symposium on Light Microscopy, issued as separate publication ASTM *STP No. 143*.

[2] These new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

[3] 1952 Book of ASTM Standards, Part 3.

In Paragraph (*b*) delete the first sentence as it is no longer applicable.

Tentative Method of Test for Bleeding of Concrete (**C 232 – 49 T**).[3]—Replace this method with the revised version appended to this report.[4]

EDITORIAL REVISION

The committee recommends the following editorial changes in two tentatives:

Tentative Method of Test for Fundamental Transverse and Torsional Frequencies of Concrete Specimens (**C 215 – 52 T**).[3]—Editorially revise to insert the word "Dynamic" in six places in Section 6.

(*1*) Before "Young's" in line 1 of Section 6(*a*),

(*2*) Before "E" in the first equation,

(*3*) Before "E" and before "Young's" in the definition of "E,"

(*4*) Before "Modulus" in line 1 of Section 6(*b*),

(*5*) Before "G" in the second equation, and

(*6*) Before "G" and before "modulus" in the definition of "G."

Tentative Method of Testing Air-Entraining Admixtures for Concrete (**C 233 – 52 T**):[3]

Editorially revise as follows.—*Section 2(c), last sentence:*—Delete the word "commercially."

Footnote 4.—Delete the word "neutralized" and add the following sentences to the footnote:

> "Neutralization may be accomplished by treating 100 parts of the Vinsol resin with 9 to 15 parts of NaOH by weight. In an aqueous solution, the ratio of water to the resinate shall not exceed 12:1 by weight."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions of a standard as set forth below, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Standard Methods of Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (**D 289 – 46**):[3]

Section 5(*a*).—Add to the table the following:

Grading	Passing Sieve	Retained On Sieve	Percentage of Sample
E	⅜-in.	No. 4	50
	½-in.	⅜-in.	50

This method is under joint jurisdiction of Committees C-9 and D-4 on Road and Paving Materials and both committees have agreed upon this recommendation.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Method of Test for Coal and Lignite in Sand (**C 123 – 44**) be withdrawn and replaced by the Tentative Method of Test for Lightweight Pieces in Aggregate as appended hereto.[4]

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Specifications for Waterproof Paper for Curing Concrete (**C 171 – 49 T**),[3] which have stood without revision for four years, be adopted as standard.

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives, which have stood without revision for two or more years be retained as tentative:

Tentative Methods of Test for:

Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (**C 88 – 46 T**),

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[4] The revised method was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

Air Content (Volumetric) of Freshly Mixed Concrete (**C 173 - 42 T**),
Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (**C 234 - 49 T**), and
Soft Particles in Coarse Aggregates (**C 235-49 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

SUBCOMMITTEE ACTIVITIES

The work of Committee C-9 is carried out by three groups of subcommittees: Group I, Administration, Fred Hubbard, chairman; Group II, Research, A. T. Goldbeck, chairman; and Group III, Specifications and Test Methods, Stanton Walker, chairman. All the subcommittees have been active during the year, and many of those that have not been responsible for recommendations included in this report may be expected to have recommendations in the near future.

This report has been submitted to letter ballot of the committee, which consists of 99 voting members, plus 6 honorary members having voting privileges; 91 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. W. TELLER,
Chairman.

BRYANT MATHER,
Secretary.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specification for:

Lightweight Aggregates for Concrete Masonry Units (C 321 - 53 T), and
Lightweight Aggregates for Structural Concrete (C 130 - 53 T).

These recommendations were accepted by the Standards Committee on December 16, 1953, and the new tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-11

ON

GYPSUM*

Committee C-11 on Gypsum met on June 24, 1952, in New York, N. Y., and on March 3, 1953 in Detroit, Mich.

The committee now consists of 20 members, of whom 9 are classified as producers, 3 as consumers, and 8 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-11, jointly with Committee C-7 on Lime, presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specifications for Sand for Use in Plaster (C 35 - 39) be revised and reverted to tentative status. This recommendation was accepted by the Standards Committee on September 5, 1952, and the tentative specifications appear in the 1952 Book of ASTM Standards, Part 3, bearing the new title Tentative Specifications for Inorganic Aggregation for Use in Interior Plaster (C 35 - 52 T).

The committee is now in process of developing proposed specifications for gypsum formboards for poured gypsum concrete roof deck constructions, and gypsum concrete for reinforced poured gypsum concrete roof deck constructions.

The committee is also considering proposed revisions to the plaster aggregate specification C 35 - 52 T.[1]

This report has been submitted to letter ballot of the committee, which consists of 20 members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

L. H. YEAGER,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28-July 3, 1953.

[1] See Editorial Note.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Gypsum Formboard (C 318 - 53 T).

Revision of Tentative Specification for:

Inorganic Aggregates for Use in Interior Plaster (C 35 - 52 T).*

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

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* Jointly with Committee C-7 on Lime.

REPORT OF COMMITTEE C-12
ON
MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the past year: on June 27, 1952, in New York, N. Y., and on March 6, 1953, in Detroit, Mich.

At the present time the committee consists of 38 members, of whom 16 are classified as producers, 11 as consumers, and 11 as general interest members. There are 3 consulting members.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-12 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Specifications for Mortar for Unit Masonry (C 270 – 51 T) be revised. This recommendation was accepted by the Standards Committee on September 5, 1952, and the revised specifications appear in the 1952 Book of ASTM Standards, Part 3.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be retained without revision:

Tentative Specifications for:

Mortar for Reinforced Brick Masonry (C 161 – 44 T), and

Mortar for Unit Masonry (C 270 – 52 T)

The committee has under consideration recommendations for further revision in C 270 – 52 T. Specification C 161 – 44 T is being continued as tentative pending further study in relation to specification C 270 – 52 T.

EDITORIAL CHANGE

The committee recommends the following editorial change in the Tentative Specifications for Mortar for Unit Masonry (C 270 – 52 T):

Table I.—Change title to read "Compressive Strength of Cubes for Mortar Types."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (F. O. Anderegg, chairman) has been active in the study of mortars for earthquake-resistant construction and of pointing mortars. Pointing mortar specifications have been set up in preliminary form and referred to Subcommittee III for study.

Subcommittee IV on Specifications for Aggregates for Mortars (Stanton Walker, chairman) is reviewing the Tentative Specification for Aggregate for Masonry Mortar C 144 – 52 T.

Working Subcommittee on Efflorescence (P. L. Rogers, chairman) has been actively studying the use of efflorwicks for determining efflorescence. A paper embodying the results of cooperative studies is being prepared for publication by the chairman.

This report has been submitted to letter ballot of the committee, which consists of 38 members; 34 returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. HARDESTY,
Chairman.

JAMES A. MURRAY,
Secretary.

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*Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3 1953.

REPORT OF COMMITTEE C-14

ON

GLASS AND GLASS PRODUCTS[*]

Committee C-14 held two meetings during the past year: on October 9, 1952, at Bedford Springs, Pa., and on April 29, 1953, at New York, N. Y.

The committee, as of April 29, consisted of 46 voting members, 9 non-voting members, and 15 consulting members. The voting membership consists of 15 producers, 16 consumers, and 15 general interest members.

REVISION OF TENTATIVES

The committee recommends the following revisions of the Tentative Method of Sampling Glass Containers (**C 224 - 49 T**)[1] be adopted by the Society. The revision was approved by Committee C-14 by the following letter ballot vote: affirmative, 27; negative, 0; not voting, 7.

Table I.—Delete the table and accompanying footnotes and substitute the accompanying Table I.

Section 4(b).—Delete the last five sentences beginning with "The maximum number of cases to be sampled from any lot shall be equal, etc." and substitute the following:

> The maximum number of cases to be sampled from any lot shall equal the total number of containers to be evaluated in both the original and subsequent samplings, or one container per case. The minimum number of cases to be selected shall be (*1*) determined by use of Military Standard 105 A[2] or (*2*) that number required to

TABLE I.—MINIMUM NUMBER OF SPECIMENS FOR SAMPLE.

Classification (Test or Examination to be made on Specimen)	Continuous Production (Lehr)		Carload or Pile, Minimum Number of Containers
	Minimum Number	Time Schedule	
Annealing..........	3[a]	3 hr or less	20
Internal Pressure and Thermal Shock....	1 round[b]	3 hr or less[c]	50[d]
Visible Characteristics.	not applicable[e]	not applicable[e]	[f]
Dimensional characteristics........	1 round	variable[g]	50[h]

[a] The three containers shall represent both sides and center of lehr.

[b] A "round" is one container from each mold or mold cavity of the forming machine. Test to be made on pieces which have passed visual inspection.

[c] This time schedule required for pressure ware only.

[d] For a pass test (attribute testing): 50 containers, randomly selected, but representative of the mold cavities present in the lot, shall be tested as the first sample in a double sampling method, followed, when necessary, by a second sample of 100 containers equally random and representative of all mold cavities present. The acceptance and rejection number shall be chosen from Table VI-J of Military Standard 105 A[2] according to the Acceptable Quality Level agreed upon[f]. Progressive tests (variable testing) shall be based on a single sample of 50 containers. The Standard Method of Internal Pressure Test on Glass Containers (ASTM Designation: C 147)[1] and the Standard Method of Thermal Shock Test on Glass Containers (ASTM Designation: C 149)[1] shall be followed for test details.

[e] For this category a sampling procedure normally is unnecessary since 100 per cent inspection is usually carried out.

[f] When the intention of the sampler is to classify the ware into acceptable and non-acceptable categories, it is recommended that sampling agreements be made between purchaser and manufacturer on the basis of Military Standard 105 A[2]. This method recommends sample sizes on the basis of size of a lot and expected frequency of occurrence of objectionable deficiencies.

[g] Because of the great variation in sizes and types and variations in requirements among the various characteristics involved, a definite schedule is impractical. Timing will depend on the degree of control necessary for the characteristics involved. Typical dimensions are height, diameter, weight, capacity, lettering or decoration (position and size), and the principal dimensions of the finish (closure holding contours).

[h] Fifty containers shall be taken as a first sample, and 100 containers as a second sample, following the principle of Table VI-J of Military Standard 105 A[2] for attribute gaging. A minimum sample of one from each mold cavity represented in the lot shall be taken for measurement by variables.

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[*] Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

[2] Military Standard—Sampling Procedures and Tables for Inspection by Attributes, MIL-STD-105 A, 11 September 1950, United States Government Printing Office, Washington, D. C.

provide the number of individual containers to be evaluated, whichever quantity represents the larger number of cases. If more than one independent evaluation can be performed on the same container, no additional sample is required for the multiple evaluation.

(*1*) Minimum number of cases determined by the use of Military Standard 105 A:[2] Determine the number of cases in the load or pile and use this number as "lot size" for Table III of Military Standard 105 A.[2] Find the corresponding sample size code letter (normally column II) of this Table III. Using the first and second columns of Table IV-A, find the sample size corresponding to this code letter. This sample size will designate the number of cases to be selected at random from the load or pile. As far as possible, an equal number of individual containers shall be taken for evaluation from each case selected.

(*2*) Minimum number of cases determined by the number of individual containers to be evaluated:

In the event that (*1*) does not provide the number of containers required for the evaluation, sufficient cases shall be selected at random to provide the necessary number of containers.

Example of minimum sampling using Military Standard 105 A:

A lot of 1000 cases of 24 containers each would call for sample size code letter K (Column II, Table III), corresponding to 110 cases (Table IV-A). The minimum sample size for visible characteristic determinations for the original lot of 24,000 containers is designated by sample size code letter O. Turning to Table VI-0, it can be seen that 450 containers (approximately 4 per case selected) are needed for a single sample, 300 (approximately 3 per case) followed by 600 (approximately six per case) for the respective first and second samples for double sampling, and 100 (approximately 1 per case) for each step of multiple sampling. The containers sampled may be used to determine the relative frequency of occurrence of containers from individual mold cavities. Containers which have passed the visual examination may be used to provide samples (chosen at random from among the cases) for the annealing, internal pressure, thermal shock, and dimensional evaluations as required.

EDITORIAL CHANGE

The committee recommends the following editorial changes in the Standard Method of Flexure Testing of Glass (Determination of Modulus of Rupture) (**C 158 – 43**).[1]

Section 1.—Add the following footnote:

"The applied stress or 'fiber stress' required to break any glass specimen depends, among other factors, upon previous heat treatment, distribution if inherent stress, the effective duration of applied stress, surface treatment, and the condition of the surface. The condition of the surface is especially important. It often happens that manufactured articles of glass as distinguished from specimens of a simple shape, such as rods or laths prepared in a laboratory, will have a different surface condition depending on their mode of manufacture or the extent of scratches and abrasions inflicted subsequent to manufacture. Pristine fire-polished surfaces, pristine molded surfaces, mechanically polished surfaces, heat-treated and chemically treated surfaces all may have characteristically different breaking stresses, and these in turn may be greatly modified by the presence of scratches and abrasions accidentally inflicted. In the case of laths, the edge finish used has a pronounced influence on the breaking stress."

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Methods of Chemical Analysis of Soda-Lime Glass (**C 169– 43**)[1] and accordingly asks for the necessary nine-tenths vote for the recommendation to be submitted to letter ballot vote of the Society:

Sections 32 and 33.—Change the routine procedure for determination of Boric Oxide to read as follows:

32. *Reagents:* (*a*) *Hydrochloric Acid* (*approximately 0.1 N*).—Prepare an approximately 0.1 *N* HCl solution free of CO$_2$.

(*b*) *Sodium Hydroxide* (*approximately 6 N*).—Prepare an approximately 6 *N* NaOH solution free of CO$_2$ and B$_2$O$_3$. Solutions of NaOH attack borosilicate glass. Consequently all such solutions should be freshly prepared, or stored in bottles containing no boron.

(*c*) *Sodium Hydroxide* (*approximately 1 N*).—Dilute a measured quantity of the above 6 *N* NaOH with 5 parts of CO$_2$-free distilled water.

(*d*) *Sodium Hydroxide* (*0.05 N*).—Prepare an approximately 0.05 N NaOH solution free of CO_2, and standardize against the National Bureau of Standards standard sample 84c of acid potassium phthalate.

(*e*) *Mannite.*—Reagent grade, neutral.

33. *Procedure.*—(*a*) Intimately mix 1.000 g of the prepared sample (Section 4) and 2 to 6 g of Na_2CO_3 as preferred, in a suitable platinum dish or platinum crucible and heat at 1000 to 1100 C until a clear melt is obtained. Rotate dish or crucible so that the melt solidifies in a thin layer. Place crucible, if used, in a 250-ml beaker. Add to the beaker or dish 5 ml of diluted HCl (1:1) for each gram of Na_2CO_3 used as a flux, cover, and allow to digest at room temperature with occasional stirring with a platinum rod until the melt is disintegrated. Transfer the solution to the 250-ml beaker. Rinse the dish or crucible with cold water and dilute to 80 to 100 ml.

(*b*) Adjust to a pH between 5.0 and 5.5 with 6 N and 1 N NaOH and 0.1 N HCl, using a pH meter or a suitable indicator (such as bromcresol purple, methyl red, etc.). Digest on a hot plate or steam bath below boiling temperature (65 to 95 C) for 20 to 30 min., making sure that the pH remains in the 5.0 to 5.5 range throughout the digestion by further additions of 0.1 N HCl if necessary. A stream of CO_2-free air should be bubbled through the solution during this digestion period to aid in eliminating CO_2 and to prevent bumping. Filter through a coarse paper, using suction if desired, and collect the filtrate in a 400-ml beaker. Transfer the residue to the paper and wash the residue and paper 10 or 12 times with hot distilled water.

(*c*) Cool the solution to room temperature and determine the pH with a pH meter. Add sufficient standard 0.1 N HCl or 0.05 N NaOH from a buret, with continual stirring, until a stable pH of 5.4 is obtained. Record the buret reading. Add 40 g of mannite to the solution, which, at this stage, should have a volume between 200 and 250 ml and continue the addition of 0.05 N NaOH until pH 6.8 is reached.

(*d*) *Calculation.*—The volume of NaOH required between pH 5.4 and pH 6.8 (Paragraph (*c*)) provides a measure of the boric oxide contained in the sample. The boric oxide content of the sample shall be calculated as follows:

$$B_2O_3\text{, per cent} = 3.482\frac{VN}{W}$$

where:

V = milliliters of standard NaOH required for titration,

N = normality of the standard NaOH solution, and

W = grams of sample.

(*e*) *Blank Titration.*—The percentage of B_2O_3 shall be corrected by a blank on the reagents. This may be done conveniently by repeating the entire procedure (Paragraphs (*a*) to (*d*)) with a glass (such as the National Bureau of Standards' Standard Sample No. 80) known to contain no boron.

(*f*) The above procedure is satisfactory for the analysis of glasses containing not more than 2 per cent of boric oxide. In the analysis of glasses high in boron, use a smaller sample, a stronger NaOH standard, or both. In that case, a correction factor must be inserted in the equation under "Calculations" (Paragraph (*d*)). This correction factor should be determined by each analyst himself based on his analysis of National Bureau of Standards' Standard Sample No. 93 (corrected for ignition loss).

(*g*) *Accuracy.*—The accuracy of the procedure is as follows: from 0.1 to 2.0 per cent B_2O_3, ±0.03 to 0.05 per cent; from 10 to 20 per cent B_2O_3, ±0.10 per cent.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman) has been working on revisions of the glossary of glass terms. These revisions are a result of work on softening, strain, and annealing point carried on by Subcommittee IV on Physical and Mechanical Properties.

Subcommittee II on Chemical Analysis (F. W. Glaze, chairman) has presented the revised method for determination of B_2O_3 in soda-lime glass. Work is currently being completed on a new procedure for determination of lime and magnesia in glass sand.

Subcommittee III on Chemical Properties (J. F. Greene, chairman) has under consideration specifications for water to be used in glass durability testing.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Physical and Mechanical Properties (H. R. Lillie, chairman) has established a task group for working on proposed methods for measuring softening, strain, annealing points, and expansion of glass. A second task group is studying the flexure testing of glass.

Subcommittee VII on Glass Containers (K. C. Lyon, chairman) is currently studying revisions and modification of the Tentative Method of Sampling Glass Containers (C 224 – 49 T).

This report has been submitted to letter ballot of the committee, which consists of 47 members; 18 members returned their ballots, of whom 16 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. G. GHERING,
Chairman.

F. V. TOOLEY,
Secretary.

REPORT OF COMMITTEE C-15
ON
MANUFACTURED MASONRY UNITS*

Committee C-15 held two meetings during the past year: on June 26, 1952, in New York City, in conjunction with the Annual Meeting of the Society; and on March 4, 1953, in Detroit, Mich., during the ASTM Committee Week.

At the present time Committee C-15 consists of 67 members, of whom 30 are classified as producers, 20 as consumers, and 17 as general interest members.

A special study committee has been appointed to study the merits of a type of structural clay products unit known as a "brick-block" unit, and the advisability of preparing an ASTM standard for such intermediate clay masonry units, which are not now covered in either brick or structural clay tile standards.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revisions of Tentative Specifications for:

Glazed Masonry Units (C 126 - 52 T),
Structural Clay Facing Tile (C 212 - 52 T),
Chemical Resistant Units (C 279 - 51 T).

Tentative Revisions of Standard:

Definitions of Terms Relating to Structural Clay Tile (C 43 - 50), and
Specifications for Vitrified Clay Filter Block (C 159 - 51).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the revised tentatives, together with the tentative revisions of the standards, appear in the 1952 Book of Standards, Part 3.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Drain Tile (C 4 - 50 T), which have stood without revision for two or more years, be retained as tentative.

EDITORIAL REVISIONS

The committee recommends the following editorial changes:

Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick and Solid Masonry Units **(C 126 - 50 T)**:[1]

Tables II, III, IV, V, and VI.—Change headings from "Standard Dimension" to "Specified Dimension."

The following specifications under the jurisdiction of Committee C-15 have been approved as American Standards by the American Standards Association during the past year:

Standard Specifications for:

Concrete Building Brick, ASTM C 55 - 52 (ASA A75.1—1953),
Building Brick (Solid Masonry Units Made From Clay or Shale), ASTM C 62 - 50 (ASA A98.1—1953),

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29-July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

Facing Brick (Solid Masonry Units Made from Clay or Shale), ASTM C 216 - 50 (ASA A99.1—1953),
Structural Clay Load-Bearing Wall Tile, ASTM C 34 - 52 (ASA A74.1—1953),
Structural Clay Non-Load-Bearing Tile, ASTM C 56 - 52 (ASA A76.1—1953),
Structural Clay Floor Tile, ASTM C 57 - 52 (ASA A77.1—1953)
Solid Load-Bearing Concrete Masonry Units, ASTM C 145 - 52 (ASA A81.1—1953),
Hollow Load-Bearing Concrete Masonry Units, ASTM C 90 - 52 (ASA A79.1—1953), and
Hollow Non-Load-Bearing Concrete Masonry Units, ASTM C 129 - 52 (ASA A80.1—1953).

Methods of Sampling and Testing:

Structural Clay Tile, ASTM C 112 - 52 (ASA A83.1—1953), and
Concrete Masonry Units, ASTM C 140 - 52 (ASA A84.1—1953)

This report has been submitted to letter ballot of the committee, which consists of 67 members; 54 members have returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards a tentative revision of the Standard Specifications for Structural Clay Load-Bearing Wall Tile (C 34 - 52). This recommendation was accepted by the Standards Committee on September 9, 1953, and the tentative revision appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the past year: at Hot Springs, Va., on October 27–29, 1952, and at Detroit, Mich., on March 2–4, 1953.

The committee, as of March 31, 1953, consisted of 63 voting members, 25 nonvoting members, 2 honorary members, and 1 consulting member. The voting membership is classified as 29 producers, 17 consumers, and 17 general interest members.

A request for a change of scope of Committee C-16 has been submitted to the Board of Directors of ASTM. The principal change is an expansion to include protective coatings used in connection with insulating materials. When approved, the scope of Committee C-16 will read:

> "Formulation of test methods and specifications relating to thermal insulating materials, except those used in the insulating refractory field, and any protective coating material associated therewith to preserve their thermal insulating efficiency."

A new subcommittee will be established immediately upon approval of the new scope.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Cellular Asbestos Paper Thermal Insulation for Pipes (C 298 - 52 T),
Laminated Asbestos Thermal Insulation for Pipes (C 299 - 52 T), and
Mineral Wool Pipe Insulation; Molded Type (Low Temperatures) (C 300 - 52 T).

Tentative Methods of Test for:

Density of Preformed Pipe Covering Type Thermal Insulation (C 302 - 52 T),
Density of Preformed Block Type Thermal Insulation (C 303 - 52 T), and Vibration Resistance of Preformed Thermal Insulation for Pipes (C 304 - 52 T).

The tentative specifications were accepted by the Standards Committee on September 5, 1952; the tentative methods were accepted on December 12, 1952. All appear in the 1952 Book of ASTM Standards, Part 3.

NEW TENTATIVE

Committee C-16 recommends the publication as tentative of the following:

Tentative Recommended Practice for:

Clearance of Preformed Thermal Pipe Insulation.[1]

TENTATIVES CONTINUED WITHOUT REVISION

The following tentatives are undergoing revision in the subcommittees. Committee C-16, therefore, recommends that they be continued in their present form until changes are completed:

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

Tentative Specification for:

Mineral Wool Batt Insulation (Industrial Type) (C 262 - 51 T),
Mineral Wool Blanket Insulation (Metal Mesh Covered) (C 263 - 51 T),
Mineral Wool Felt Insulation (Industrial Type) (C 264 - 51 T), and
Mineral Wool Blanket Type Pipe Insulation (C 280 - 51 T).

Tentative Method of Test for:

Water Vapor Permeability of Sheet Materials Used in Connection with Thermal Insulation (C 214 - 48 T), and
Thermal Conductance and Transmittance of Built-Up sections by Means of The Guarded Hot Box (C 236 - 49 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-I on Block and Pipe Insulation (W. D. Stevens, chairman) is balloting or reviewing proposed specifications for mineral wool thermal insulating board for roofs; mineral wool block or board for elevated temperatures; mineral wool block or board for low temperatures; cellular glass insulating block; calcium silicate block and pipe covering; and corkboard. The subcommittee is also reviewing the Tentative Specification for Mineral Wool Molded Pipe Insulation (C 281 - 51 T) and Standard Method of Test for Flexural Strength of Preformed Block Type Thermal Insulation (C 203 - 51).

Four proposed specifications covering 85 per cent magnesia and diatomaceous earth thermal insulation in the block and pipe forms are being reviewed following committee letter ballot.

Subcommittee S-II on Structural Insulating Board (R. R. Sullivan, chairman) has completed a racking load test for structural insulating board and sheathed wall panels and is developing methods of test for determining flame resistance of structural insulating board and for thickness of insulating board. The subcommittee is reviewing the Standard Specification for Structural Insulating Board made from Vegetable Fibers (C 208 - 48) and the Standard Methods of Testing Structural Insulating Board made from Vegetable Fibers (C 209 - 48).

Subcommittee S-III on Insulating Cement (J. E. Gaston, chairman).—Proposed tentative methods of test for adhesion of dried thermal insulating cement and for compressive hardness of insulating cement are being processed through the Significance of Test Subcommittee. Consideration is being given to a method of test for measuring wet adhesion of thermal insulating cement.

Subcommittee S-IV on Blanket Insulation (H. E. Lewis, chairman) is developing a method of testing combustibility of blanket type thermal insulations and is working in cooperation with Subcommittee S-II on details of a proposed comparative combustibility test procedure. The Tentative Specification for Mineral Wool Felt Insulation (Industrial Type) (C 264 - 51 T) is being reviewed by the subcommittee.

Subcommittee S-V on Loose Fill Insulation (G. H. Knode, chairman) is developing a method of test for determining the density at which loose fill insulation should be applied.

Subcommittee T-VI on Thermal Conductivity (J. D. Babbitt, chairman) has completed revisions of the Tentative Method of Test for Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 236 - 49 T), and a new tentative method of test for thermal conductivity of pipe insulation which will be submitted to Committee C-16 for letter ballot. The subcommittee has completed a significance of test statement for the guarded hot plate method (C 177 - 45) which will be submitted to Committee C-16 for letter ballot.

Subcommittee T-VII on Special Thermal Properties (G. W. Rapp, chairman) is continuing its development of methods of test for determining specific heat, maximum use temperature limit, and emissivity.

Subcommittee S-VIII on Dimensional Standards (H. P. Hoopes, chairman) has been assigned the development of a tentative method of sampling thermal insulation.

Subcommittee T-IX on Vapor Transmission (R. H. Heilman, chairman).—Proposed definitions pertaining to water vapor transmission have been submitted to Committee C-16 for letter ballot. The subcommittee approved revisions of a tentative method of test for water vapor permeability for thick materials for letter ballot.

Task Group on Physical Properties of Vapor Barriers Other Than Permeance (H. E. Rapp, chairman) is developing methods of testing physical properties of vapor barriers and at the present time is considering flame propagation in connection with plastic types.

Task Group for the Determination of Moisture Vapor Measurement Units (Ray Thomas, chairman).—The Task Group of Subcommittee T-IX charged with the responsibility for the determination of moisture vapor measurement units is conducting tests on various films for correlating purposes, and tests will be continued on film type materials and thicker units of insulating materials.

Research Project.—A research project to determine the effect of moisture on thermal conductivity has been set up at The Pennsylvania State College and work will be started in the near future.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members, of whom 47 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. R. Queer,
Chairman.

W. L. Gantz,
Secretary.

Editorial Note

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

85 Per Cent Magnesia Thermal Block Insulation (C 319 – 53 T), and
85 Per Cent Magnesia Thermal Pipe Insulation (C 320 – 53 T).

Tentative Revision of:

Standard Definition of Terms Relating to Thermal Insulating Materials (C 168 – 51).

The new tentative specifications were accepted by the Standards Committee on November 9, 1953, and the tentative revision was accepted on September 9, 1953; the three recommendations appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-17

ON

ASBESTOS-CEMENT PRODUCTS*

Committee C-17 on Asbestos-Cement Products and its subcommittees held a meeting on March 5, 1953, at the time of the 1953 Committee Week of the Society in Detroit, Mich.

The membership of the committee consists of 30 voting members, composed of 11 producers, 3 consumers, and 16 general interest members.

REVISION OF TENTATIVE

After more than two years of study and investigation, Committee C-17 recommends that the Tentative Specification for Flat Asbestos-Cement Sheets (**C 220 – 49 T**) be revised as appended hereto.[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee has agreed upon proposed revisions and letter ballots are being processed on the following tentatives. They are therefore recommended for continuation without change:

Tentative Specifications for:

Asbestos-Cement Corrugated Sheets (C 221 – 49 T),[2]

Asbestos-Cement Roofing Shingles (C 222 - 49 T),[2] and

Asbestos-Cement Siding Shingles and Clapboards (C 223 - 49 T).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Test (C. R. Hutchcroft, chairman) has spent much time in evaluating the methods of test that are incorporated in the proposed revisions of the current specifications and in writing the statements covering the significance of these tests. It is currently studying test methods that can determine handleability of asbestos-cement products and also searching for a way to test for organic material in these products.

Subcommittee II on Specifications (M. V. Engelbach, Chairman).—The problem confronting Subcommittee II has been to arrive at proper values for asbestos-cement products after new test procedures have been established. Agreements were reached and are being incorporated in the revised proposals.

This report has been submitted to letter ballot of the committee, which consists of 30 voting members; 21 members returned their ballots, of whom 18 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

DAVID WOLOCHOW,
Chairman.

CHESTER C. KELSEY,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] The revised tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 3.

[2] 1952 Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-17 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Asbestos-Cement Corrugated Sheets (C 221 - 49 T),
Asbestos-Cement Roofing Shingles (C 222 - 49 T),
Asbestos-Cement Siding Shingles and Clapboards (C 223 - 49 T), and
Asbestos-Cement Pressure Pipe (C 296 - 52 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the revised tentative specifications appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-19

ON

STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held three meetings during the year; one meeting was held on June 24–25, 1952, in conjunction with the Annual Meeting at New York City, the second on October 3–4, 1952, at the Massachusetts Institute of Technology, Cambridge, Mass., and the third on April 9–10, 1953, at the Chance Vought Aircraft Division of United Aircraft Corp., Dallas, Tex.

The present committee consists of 64 members with 51 voting members, of whom 23 are classified as producers, 7 as consumers, and 21 as general interest members.

ADOPTION OF TENTATIVES AS STANDARD

Committee C-19 recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Density of Core Materials for Structural Sandwich Constructions (C 271 – 51 T),[1]
Water Absorption of Core Materials for Structural Sandwich Constructions (C 272 – 51 T)[1],
Shear Test of Sandwich Constructions in Flatwise Plane (C 273 – 51 T),[1] and
Terms Relating to Structural Sandwich Constructions (C 274 – 51 T).[1]

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties of Basic Materials (George Gerard, chairman).—R. C. Platow resigned as chairman in March, 1953, and George Gerard accepted appointment as chairman.

The present tentatives C 271 – 51 T and C 272 – 51 T have been reviewed and are recommended for adoption as standard, as noted earlier in this report. Several methods which will be subjected to letter ballot in the near future include core compression tests, core shear tests, core delamination tests, thickness tests, and possible heat distortion tests.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction (A. G. H. Dietz, chairman).—The present tentative C 273 – 51 T has been reviewed and recommended for adoption as standard, as noted earlier in this report. Several test methods are being considered for determining peel strength. Other test methods which may be circulated for letter ballot soon consider compression and flexure of sandwich constructions. The tension flatwise test, currently tentative C 297 – 52 T, will be reviewed in light of the comments received from Committee E-1.

Subcommittee III on Permanence, Durability, and Simulated Service (J. H. Gibbud, chairman) is considering possible exposure of specimens at ASTM test sites. A fatigue test for sandwich constructions or core materials is being considered. The design of an outdoor exposure test cubicle will be continued.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Nomenclature and Definitions (D. G. Reid, chairman).—The present tentative C 274 - 51 T has been reviewed and recommended for adoption as standard, as noted earlier in this report. Additional definitions relating to core material types and forms are being considered. A letter ballot on these will be conducted in the near future.

This report has been submitted to letter ballot of the committee, which consists of 51 voting members; 37 returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

Edward W. Kuenzi,
Chairman.

B. D. Beamish,
Secretary.

REPORT OF COMMITTEE C-21
ON
CERAMIC WHITEWARE*

Committee C-21 held three meetings during the year: June 27, 1952 and April 27, 1953 in New York City, and September 25, 1952 in Bedford Springs, Pa. A Symposium on Test Methods for Process Control in Ceramic Whitewares was presented at the 1952 Annual Meeting of the Society.

The present voting membership consists of 47 producers, 10 consumers, and 19 general interest.

There are at present under consideration revisions of several terms and definitions now published as tentative under the designation C 242 – 50 T.[1] All of the definitions are being recommended for continuation as tentative without change, pending revision or recommendations by the committee for approval as standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman).—At this group's several meetings during the year, the major activity dealt with the development of terms and definitions for degrees of vitrification. This has proved difficult because differing usages for certain terms have been adopted by various branches of the whiteware industry.

Subcommittee II on Tests and Specifications (Van E. Campbell, chairman).—Five methods of test for clays, developed in the subsection on clays (G. W. Phelps, chairman) and three product tests developed by the subsection on fundamental properties (W. C. Mohr, chairman) are in the final stages of completion as proposed tentatives. This subsection is active also in the development of test procedures for water absorption and moisture expansion.

Subcommittee III on Research (H. Z. Schofield, chairman).—This subcommittee investigated the factors involved in obtaining comparable and reproducible values for modulus of rupture in bending, and conducted an investigation to determine sub-sieve particle sizes.

This report has been submitted to letter ballot of the committee, which consists of 76 voting memberships; 62 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

C. G. HARMAN,
Chairman.

R. F. GELLER,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-21 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Sampling Whiteware Clays (C 322 - 53 T),
Chemical Analysis of Whiteware Clays (C 323 - 53 T),
Test for Free Moisture Content of Whiteware Clays (C 324 - 53 T),
Test for Wet Sieve Analysis of Whiteware Clays (C 325 - 53 T),
Test for Drying and Firing Shrinkages of Whiteware Clays (C 326 - 53 T),
Test for Linear Thermal Expansion by the Interferometric Method (C 327 - 53 T),
Test for Modulus of Rupture of Fired Dry-Pressed Whiteware Specimens at Normal Temperature (C 328 - 53 T), and
Test for True Specific Gravity of Fired Whiteware Materials (C 329 - 53 T).

Tentative Method C 323 was accepted by the Standards Committee on January 13, 1954, and the remaining seven methods were accepted on December 16, 1953. They all appear in Part 3 of the 1953 Supplement to Book of ASTM Standards.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held one meeting during the year on October 10–11, 1952, as guests of Battelle Memorial Inst., Columbus, Ohio. The spring meeting normally held by the committee was deferred until the Annual Meeting of the Society at which time the committee will sponsor a symposium on "Porcelain Enamels and Ceramic Coatings as Engineering Materials."

During the past year one new member has been added to the committee and there have been two resignations. At the present time, the committee consists of 36 members, of whom 30 are voting members, with 16 classified as producers, 5 as consumers, and 9 as general interest.

NEW TENTATIVES

Committee C-22 recommends that the following two methods be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:

Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal, and
Warpage of Porcelain Enameled Flatware.

ADOPTION OF TENTATIVE AS STANDARD

Committee C-22 recommends that the Tentative Method of Test for Acid Resistance of Porcelain Enamels (Room Temperature Test) (**C 282 – 51 T**)[2] be approved without change for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives which have stood for two years without revision be continued as tentatives:

Tentative Methods of Test for:

Resistance of Porcelain Enameled Utensils to Boiling Acid (**C 283 – 51 T**), and
Impact Resistance of Porcelain Enameled Utensils (**C 284 – 51 T**)

Tentative Definitions of:

Terms Relating to Porcelain Enamel (**C 286 – 51 T**).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (L. S. O'Bannon, chairman) is sponsoring the symposium on "Porcelain Enamels and Ceramic Coatings as Engineering Materials" referred to earlier in the report. Work on the survey of problems involving thermal shock and the effect of porcelain enamel coatings on the fatigue of metals at high temperatures is being continued.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

[2] 1952 Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Nomenclature (E. E. Howe, chairman) is working on the revision of the Tentative Definitions of Terms (C 286 – 51 T).

Subcommittee III on Tests and Specifications (R. F. Bisbee, chairman).—Sections 1 and 2, Raw Materials and Materials in Process (W. A. Deringer, chairman), are preparing a proposed test method for determination of reflectivity and coefficient of scatter for ballot by the subcommittee. The sections are investigating seven different problems, chiefly concerning raw materials.

Section 3, Finished Products (J. H. Gregory, chairman), is studying nine porcelain enamel properties preparatory to preparing tentative test methods therefor. During the past year the section completed and passed to letter ballot proposed tentative test methods for the determination of warpage and adherence as noted earlier in this report. The proposed Tentative Method of Test for Reflectance of Opaque Specimens, which was approved by letter ballot of Committee C-22, has been submitted to Committee E-12 on Appearance Properties in order that a single test method may be prepared to include the above proposed method and a standard method (D 771) of Committee D-1.

This report was submitted to letter ballot of the committee, which consists of 30 voting members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: in New York, N. Y., June 23 to 25, 1952, in connection with the 1952 Annual Meeting of the Society; and in Detroit, Mich., March 2 to 4, 1953, in connection with the Spring Group Committee Meetings.

At the June, 1952, meeting, Committee D-1 celebrated the 50th anniversary of its founding. The occasion was marked by a golden anniversary banquet on June 24, and a special session at which the major accomplishments of its 50 years of progress were reviewed.[1] The arrangements for the 50th anniversary celebration were handled by a committee under the chairmanship of J. C. Moore. The special session for the presentation of papers, covering the history of paint testing, was arranged by the Papers Committee, under the chairmanship of W. H. Lutz.

At the March, 1953, meeting, N. P. Beckwith presented an illustrated talk on "The Production Man's View of the Technical Man's Responsibilities." For the June, 1953, meeting, it is planned to present a companion paper on the responsibility to management of technical men in the paint industry.

Cooperative work with the Federation of Paint and Varnish Production Clubs has continued active during the year under the Joint Committee (S. Werthan, chairman). The number of standards approved is now 52. In addition, the following ASTM methods have been approved in the past year by the Standards and Methods of Test Committee of the Federation and will be assigned Federation numbers:

Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 46),
Methods of Chemical Analysis of White Linseed Oil Paints (D 215 - 41),
Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 51),
Methods for Chemical Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate, and Chromium Oxide Green (D 126 - 50 T),
Methods for Chemical Analysis of Blue Pigments (D 1135 - 50 T),
Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (D 185 - 45), and
Methods of Testing Para Red and Toluidine Red Pigments (D 970 - 51).

Nine additional ASTM test methods are now under study by the Federation. Similarly, two Federation methods are being considered within Committee D-1. The two organizations are also continuing their cooperation in the preparation of specifications for mineral spirits.

The Committee on Inter-Committee Relations, which is composed of D-1 representatives on other ASTM technical committees, is continuing its close contact with those committees whose activities are of interest to Committee D-1. Two D-1 representatives have been appointed to Subcommittee VII on

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] "Fifty Years of Paint Testing," Symposium on Paint, Am. Soc. for Testing Mats. (1952). Issued as a separate publication, *STP No. 147*.

Rubber Latices, of Committee D-11 on Rubber and Rubber-Like Materials.

The cooperative work with Committee D-2 on Petroleum Products and Lubricants is being continued. In cooperation with Committee D-16 on Industrial Aromatic Hydrocarbons, specifications for three industrial aromatic hydrocarbons have been prepared and proposed for joint sponsorship by the two committees.

The American Standards Association has approved as American Standard the ASTM Standard Specifications for Spirits of Turpentine (D 13 – 51). Committee D-1 has submitted seven additional standards under its jurisdiction for ASA approval.

Proposed Methods to be Published as Information

The committee recommends the following two proposed methods of test for publication as information, as appended hereto:[2]

Proposed Method of Test for Viscosity Reduction Power of Hydrocarbon Solvents, and

Proposed Method of Test for Flash Point of Volatile Flammable Materials by the Tag Open-Cup Apparatus, as revised.

New Tentatives

The committee recommends for publication as tentative the following specifications and methods of test, as appended hereto:[3]

Tentative Specifications for:

High-Gravity Glycerine,

Tentative Methods of:

Sampling and Testing High-Gravity Glycerine,

Test for Nonvolatile Content of Resin Solutions, and

Calculating Small Color Difference from Data Obtained on the Hunter Multipurpose Reflectometer.

Revision of Tentative

Committee D-1 recommends that the Tentative Specifications for Dehydrated Castor Oil (**D 961 – 51 T**)[4] be revised as follows:

Section 3.—In Paragraph (*a*), delete the words "Iodine Number."

Add a new Paragraph (*d*), to read as follows:

(*d*) *Iodine Number.*—The procedure described in Sections 22 to 25 of the Tentative Methods of Testing Drying Oils (ASTM Designation: D 555) shall be used to determine iodine number, except that the sample weights shall be 0.11 to 0.13 g of oil.

Tentative Revision of Standards

Methods of Testing Varnishes (**D 154 – 50**).[4]—Replace Section 5 with the following:

5. (*a*) The primary standards for color consist of solutions defined by their spectral transmittance in a 1-cm cell with parallel sides. The trichromatic coefficients of these solutions are given in Table I. These are determined on a

TABLE I.—REFERENCE STANDARD COLOR SOLUTIONS.

Color Standard Number	Trichromatic Coefficients		Potassium Chloroplatinate, g per 100 ml of sulfuric acid	Iron-Cobalt Solutions			Potassium Dichromate, g per 100 ml of sulfuric acid
	x	*y*		Ferric Chloride Solution, ml	Cobalt Chloride Solution, ml	Hydrochloric Acid, ml	
1	0.3190	0.3271	0.550	...	...	...	0.0039
2	0.3241	0.3344	0.865	...	...	...	0.0048
3	0.3315	0.3456	1.330	...	...	...	0.0071
4	0.3433	0.3632	2.080	...	...	...	0.0112
5	0.3578	0.3820	3.035	...	...	...	0.0205
6	0.3750	0.4047	4.225	...	...	...	0.0322
7	0.3927	0.4235	5.550	...	...	...	0.0384
8	0.4118	0.4432	7.185	...	...	...	0.0515
9	0.4300	0.4620	...	3.3	2.6	94.1	0.0780
10	0.4490	0.4775	...	5.1	3.6	91.3	0.164
11	0.4836	0.4805	...	7.5	5.3	87.2	0.250
12	0.5084	0.4639	...	10.8	7.6	81.6	0.380
13	0.5395	0.4451	...	16.6	10.0	73.4	0.572
14	0.5654	0.4295	...	22.2	13.3	64.5	0.763
15	0.5870	0.4112	...	29.4	17.6	53.0	1.041
16	0.6060	0.3933	...	37.8	22.8	39.3	1.280
17	0.6275	0.3725	...	51.3	25.6	23.1	2.220
18	0.6475	0.3525	...	100.0	0.0	0.0	3.00

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[2] See pp. 342 and 348.

[3] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 4.

[4] 1952 Book of ASTM Standards, Part 4.

1-cm layer of the solution according to the Standard Method of Test for Spectral Characteristics and Color of Objects and Materials (ASTM Designation: D 307).

(*b*) For purposes of comparison, the use of permanent solutions, the color of which has been determined, is more satisfactory. The approximate composition of solutions giving each of the 18 colors is also given in Table I. The solutions are made from pure crystalline potassium chloroplatinate in 0.1 *N* hydrochloric acid, or, in the darker colors, from stock solutions of ferric chloride, cobalt chloride, and hydrochloric acid, prepared as follows:

Repeat Items (*1*), (*2*), and (*3*) of present Paragraph (*b*).

(*c*) Solutions of potassium dichromate in pure sulfuric acid (sp gr 1.84) may be used as reference standards. The approximate composition of these standards is also given in Table I. Each solution must be freshly made for the color comparison, using gentle heat, if necessary, to effect solution.

NOTE 4.—Same as present Note 4.

Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (**D 333 - 40**).[4]—Revise as follows:

Section 3.—Change drying time test to read as follows:

3. (*a*) The sample to be tested and a mutually agreed upon reference standard, preferably of the same viscosity and solids content, shall be simultaneously drawn down on a horizontally held, smooth, clean, dry glass plate approximately 8 by 10 in. in dimensions, using a 6-in. blade applicator of a clearance to deposit 0.003 in. of wet film. The films preferably shall be applied and tested under conditions of 25 ± 1 C and 50 ± 5 per cent relative humidity, with no direct sunlight falling upon the film surface. After the film has set, the panels shall be placed in a vertical position in a location free from air movement. While conditions of constant temperature and humidity are preferable for this test, they are not mandatory, because of the inclusion in the test of the reference standard.

(*b*) The time of application shall be recorded and the drying time shall be judged by drawing a finger across the sample and control, or by touching or pressing the film with the finger at intervals with equal pressure at approximately the same relative locations. These intervals of time will vary with the type of lacquer being tested, and should be selected accordingly. The time of drying shall be recorded, and the total interval referred to as "drying time to touch."

Section 4.—Change the gloss test to read as follows:

4. A panel for the gloss test shall be prepared by pouring the sample and a mutually agreed-upon reference standard on an opaque, mutually agreed-upon surface in such a manner that the two materials flow together along an adjacent edge. The panel shall be examined for gloss after 24 hr. A convenient method of determining apparent gloss is to hold the test panel at an angle of incidence approaching 180 deg to a source of artificial light, and to note the comparative sharpness of the definition of the image (for example, the lamp filament). For determination of 60-deg specular gloss, the procedure described in the Tentative Method of Test for 60-deg Specular Gloss of Paint Finishes (ASTM Designation: D 523) should be followed.

Section 8.—Change nonvolatile matter test to read as follows:

8. (*a*) ***Nonvolatile Matter.***—Into a flat-bottom metal or glass dish containing a stirring rod, 4 to 6 g of the sample to be tested shall be weighed in such a manner as to minimize the loss of volatile solvents. Then 100 ml of acetone shall be added and stirred until solution of the material is complete, and 10 ml of water added slowly, while stirring vigorously. The solution shall be evaporated to dryness over a hot-water bath and then heated for 1 hr in an oven at 100 to 105 C, allowed to cool 20 min in a desiccator, and weighed on an analytical balance. (The dish should be covered in such a manner as to allow solvents to escape and to exclude contamination by dirt in the atmosphere.) If the precipitate or residue is lumpy, it shall be redissolved in the solvent combination, and precipitated again with water.

(*b*) The nonvolatile content shall be reported either in terms of per cent by weight or as grams per 100 ml, according to the option of the investigator. For the latter method of reporting, specific gravity of the lacquer under test is required. For this determination, a pycnometer may be used as described under the Standard Methods of Testing Varnishes (ASTM Designation: D 154). A slightly less accurate but acceptable procedure is as follows:[*]

Using a long-stem funnel, flow the thoroughly mixed lacquer into a weighed calibrated 100-ml volumetric flask until this is filled to a point about $\frac{1}{2}$ in. under the 100-ml mark; the use of the funnel ensures that none of the lacquer will spill on the neck of the flask above the 100-ml mark. Adjust the temperature to 25 ± 1 C by use of a constant-temperature bath or room storage under conditions to produce this temperature. Add lacquer to fill to the 100-ml graduation and weigh to the first decimal place, using a torsion balance. The weight of the flask contents divided by 100 gives the specific gravity, and this value multiplied by the per cent nonvolatile by weight will give the grams solids per 100 ml lacquer.

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[*] The commonly used weight per gallon cup which contains 83.3 ml may be used for this determination when only moderate accuracy is required.

Figure 1.—Add the following note under the illustration of the Consistency Cup Apparatus:

NOTE 4.—The overflow, though desirable, is not mandatory, and other consistency cups of the described dimensions and having orifice diameters as noted are satisfactory for this determination.

Section 9.—Add the following Paragraph (*c*) to the consistency test:

(*c*) *Calibration.*—Time of efflux from consistency cups above a given minimum is proportional to kinematic viscosity in stokes:

$$V_s = KT$$

where:

V_s = kinematic viscosity in stokes,

K = conversion factor to provide value in stokes when time of efflux in seconds is above a stated minimum, and

T = time of efflux in seconds.

The conversion constant, K, for the viscosity cups is as follows:[a]

Orifice, in.	Factor K	Minimum Time, sec
0.07	1.4	60
0.10	4.8	25
0.15	21	9
0.25	140	4

National Bureau of Standard Oils are recommended for use in determining the accuracy of consistency cups. Absolute viscosity in poises of these standards may be converted to stokes by the formula:

$$V_s = \frac{N}{d}$$

where:

V_s = kinematic viscosity in stokes,

N = absolute viscosity in poises, and

d = density.

A suitable correction factor may be used on any cup which does not conform to the above listed constants.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the following standards be revised as appended hereto[5] and reverted to tentative:

D 523 – 51. Method of Test for 60-deg Specular Gloss, and

D 605 – 42. Specifications for Magnesium Silicate Pigment.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-1 recommends that the following seven tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of:

Testing Drying Oils (D 555 – 51 T), with the following editorial revision: In Section 25 (b) add the following Note 2, inserting it immediately before the table, and change present Note 2 in Paragraph (*d*) to Note 3: "Note 2.—The sample weight to be used for dehydrated castor oils is given in the Tentative Specifications for Dehydrated Castor Oil (D 961 - 51 T) under 3 (*d*), Iodine Number."

Test for Roundness of Glass Spheres (D 1155 – 51 T),

Test for Heptane Number of Hydrocarbon Solvents (D 1132 – 50 T),

Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents (D 1134 – 50 T),

Testing Varnishes (D 154 – 52 T), Drying Time Test and Rosin-Pentaerythrital Ester Flexibility Test, Sections 3 to 20, inclusive,

Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test panels (D 823 – 51 T), with editorial revision changing "Procedures" to "Methods" and describing the tests in the imperative mood, and

Measurement of Dry Film Thickness of Nonmagnetic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Magnetic Base (D 1186 – 51 T).

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[a] M. R. Euverard, "Evaluation of Empirical Viscosity Measurements for Varnishes and Resin Solutions," ASTM BULLETIN, No. 169, October, 1950, p. 67.

[5] The revised tentative methods were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 4.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following method, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Methods of Sampling and Testing Lacquer Solvents and Diluents (**D 268 - 49**).[4]—Revise as follows:

Section 4.—Revise to read: "Determine color by the Tentative Method of Test for Color of Clear Liquids (Platinum-Cobalt Scale) (ASTM Designation: D 1209)."

Sections 23 to 25.—Substitute the following for present Sections 23 to 25: "Determine dimethylketone by the Tentative Method of Test for Purity of Acetone and Methyl Ethyl Ketone (ASTM Designation: D 1154)."

WITHDRAWAL OF STANDARD

The committee recommends that the Specifications for Perilla Oil, Raw or Refined (D 125 - 48) be withdrawn.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following ten tentatives be continued as tentative without revision:

Tentative Specifications for:

Asphalt-Base Emulsions for Use as Protective Coatings for Metal (D 1187 - 51 T)
Heavy Petroleum Spirits (Heavy Mineral Spirits) (D 965 - 48 T)
Methanol (Methyl Alcohol) (D 1152 - 51 T)
Methyl Isobutyl Ketone (D 1153 - 51 T)

Tentative Method of:

Testing Bituminous Emulsions for Use as Protective Coatings for Metal (D 1010 - 49 T)
Test for Color of Orange Shellac (D 29 - 41 T)
Test for Ester Value of Tricresyl Phosphate (D 268 - 49 T)
Test for Kauri-Butanol Value of Hydrocarbon Solvents (D 1133 - 50 T)
Test for Purity of Acetone and Methyl Ethyl Ketone (D 1154 - 51 T)
Water Immersion Test of Organic Coatings on Steel (D 870 - 51 T)

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (D. S. Bolley, chairman) has revised the Tentative Methods of Testing Drying Oils (D 555 - 51 T) to specify the use of the Pensky-Martens high-range thermometer in the heat and quality tests for tung oil. Work is continuing on a procedure for sampling drying oils and related materials and on determining total unsaturation by hydrogenation. A draft of proposed specifications for safflower oils is in preparation. Work has been initiated on the spectrophotometric analysis of drying oils, and procedures for determining iodine value of dehydrated castor oils. The subcommittee plans to revise the Standard Specifications for Oiticica Oil (D 601 - 46) and to prepare specifications for fatty acids.

Subcommittee III on Bituminous Emulsions (R. H. Cubberley, chairman) plans to revise the Tentative Methods of Testing Bituminous Emulsions for Use as Protective Coatings for Metal (D 1010 - 49 T), in collaboration with Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Subcommittee IV on Traffic Paint (W. G. Vannoy, chairman) plans to conduct cooperative work with the highway departments of several states; the durability tests will include road service tests, the Goodyear accelerated tests, and various other accelerated tests. Work is being continued on the crush resistance, sieve analysis, wettability, clarity, and chemical stability of glass spheres.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) is continuing its cooperation with the Federation of Paint and Varnish Production Clubs and other interested groups in the revisions of the Standard Specifications for Petroleum Spirits (Mineral Spirits) (D 235 - 39). It is planning to conduct cooperative work to determine the difference in results when Methods D 1078 and D 86 are used in distillation tests of mineral spirits.

Subcommittee VI on Definitions (G. G. Sward, chairman) is considering a definition for "silicone resin" and has tabled a proposal for a definition for "white creosote paint."

Subcommittee VII on Accelerated Tests for Protective Coatings (R. H. Sawyer, chairman) has undertaken a revision of the Standard Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 52) to include both the drop test and the contact-angle test to supplement the water-break test for cleanliness of panel surfaces. The group working on humidity testing is preparing a report. A new set of photographic standards has been prepared to replace those included in the Standard Method for Evaluating Resistance to Blistering of Paints on Metal when Subjected to Immersion or Other Exposure to Moisture or Liquids (D 714 - 45). The effect of the water source on the severity of failures under accelerated light and water exposures will be investigated.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) is bringing to a conclusion its revision of the Tentative Method of Analysis of White Pigments (D 34 - 51 T), and is actively working on a revision of the Standard Method of Chemical Analysis of Dry Mercuric Oxide (D 284 - 33).

Subcommittee IX on Varnish (J. C. Weaver, chairman) plans to take up the problem of standardizing illuminants and viewing conditions for the Gardner Color Standards as a sequel to the recommended redefinition of the standards. Work is continuing on skinning tests, drying time, driers and drier testing, and the determination of rosin. Work has been started on the analysis of phthalic anhydride, the abrasion resistance of floor varnishes, and acid numbers.

Subcommittee X on Optical Properties (M. P. Morse, chairman) prepared the proposed method of test for color difference measurements with the Hunter multipurpose reflectometer, and is continuing its work on establishing methods of test for five other commercial color measuring instruments. It is continuing its work on hiding power and has organized a group to develop a method for measuring the gloss of clear finishes over wood. Definitions for gloss, specular gloss, and luminous directional reflectance are in preparation. The consolidated gloss method has been recommended as a replacement for the present Standard Method of Test for 60-deg Specular Gloss (D 523 - 51). It has under study several revisions of the Standard Method of Test for Tinting Strength of White Pigments (D 332 - 36).

Subcommittee XI on Resins (C. F. Pickett, chairman) prepared the proposed Specifications for High-Gravity Glycerin, and Methods of Sampling and Testing High-Gravity Glycerin, and the Method of Test for Nonvolatile Content of Resin Solutions. An ultraviolet method and a chemical method for the analysis of phthalic anhydride are being prepared as tentative revisions of the Standard Methods of Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions (D 563 - 52). Work is continuing on the applicability of the foil method to heat-sensitive resins, the viscosity of brittle resins in solution, and the determination of softening point of resins.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) is continuing its work on the development of suitable methods for determining cold alcohol insoluble matter, polymerized bodies, and color of lac resins, and on the revision of the Standard Methods of Sampling and Analysis of Shellac (D 29 – 40). It is continuing its cooperation with Technical Committee 50 on Lac Resins, of the International Standards Organization.

Subcommittee XV on Specifications for Pigments Dry and in Oil (C. L. Crockett, chairman) prepared the revision of the Standard Specifications for Magnesium Silicate Pigment (D 605 – 42), and is preparing a revision of the Standard Specifications for Copper Phthalocyanine Blue (D 963 – 49). An exploratory program of cooperative work has been outlined on a method for reporting the fineness characteristics of pigments.

Subcommittee XVI on Printing Inks (M. C. Rogers, chairman) is bringing to a conclusion its work on a proposed method of test for fineness of grind and test methods for drying time and specific gravity. Progress is reported by the groups working on Definitions, Methods Review, Rubproofness, Rheology, and Paper-Ink Relations.

Subcommittee XVII on Flash Point (A. L. Brown, chairman) revised the Proposed Method of Test for Flash Point by the Tag Open Cup, published in Appendix I as information, as a result of a cooperative work program by eight laboratories using three samples of hydrocarbon solvents with flash points in the range 75 to 100 F. It is planned to test other organic solvents, particularly nonhydrocarbons, over the range of 20 to 80 F flash points. Work is under way on the development of improved equipment for determining the open-cup flash point of viscous materials.

Subcommittee XVIII on Physical Properties of Materials (M. R. Euverard, chairman) prepared the proposed revisions for the Tentative Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels (D 823 – 51 T). Progress has been reported by the groups working on oil absorption, consistency of pastes, adhesion, hardness, water vapor permeability, fire retardency of paints, and flow properties. New groups have been organized for the study of the measurement of dry film thickness of coatings applied to nonmagnetic bases and the specific gravity of paint and paint materials.

Subcommittee XXV on Cellulosic Coatings and Related Materials (F. H. Lang, chairman) prepared the revisions of tests for drying time, gloss, nonvolatile matter, and consistency in the Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 40). Additional work is in progress on a perspiration test, an improved print test, and a moisture change resistance test.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) is continuing its work on the preparation of a method of classifying ferrous surfaces for painting and the preparation of aluminum surfaces for painting. A new group has been organized to work on the development of a method of preparation of magnesium surfaces for painting.

This report has been submitted to letter ballot of the committee, which consists of 352 voting members; 216 members returned their ballots, of whom 194 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

W. A. GLOGER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Residual Odor of Lacquer Soluents and Diluents (D 1296 – 53 T), and
Test for No-Dirt-Retention Time of Traffic Paint (D 1297 – 53 T).

Revision of Tentative Methods of:

Chemical Analysis of White Pigments (D 34 – 51 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentative methods appear in the 1953 Supplement to Book of ASTM Standards, Part 4.

APPENDIX I

PROPOSED METHOD OF TEST FOR VISCOSITY REDUCTION POWER OF HYDROCARBON SOLVENTS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3 Pa.

Scope

1. This method of test describes a procedure for determining the comparative viscosity reduction power of hydrocarbon solvents for various types of resinous materials. The solvents should have an initial boiling point over 40 C (104 F) and an end boiling point below 300 C (572 F) when determined in accordance with the Tentative Method of Test for Distillation Range of Lacquer Solvents and Diluents (ASTM Designation: D 1078)[3] or the Standard Method of Test for Distillation of Gasoline, Naptha, Kerosine, and Similar Petroleum Products (ASTM Designation: D 86).[3]

NOTE.—Method D 86 is to be used to determine the initial boiling point and dry point for mineral spirits and similar petroleum solvents. Method D 1078 is to be used for pure compounds and narrow boiling range cuts.

Definition

2. *Viscosity Reduction Power* is the ratio of the viscosity of the resin dispersed in a control hydrocarbon to the viscosity of a dispersion of the same resin at the same concentration in a test hydrocarbon solvent.

Apparatus

3. (*a*) *Glass Bottles*, narrow mouth, 16-oz, with corks to fit; wide mouth, 16-oz with screw cap and aluminum foil seal.

NOTE.—If desired, 1-pt friction top cans may be substituted.

(*b*) *Balance* with weights, sensitivity of 0.05 g.

(*c*) *Mortar and Pestle*, or other suitable equipment for grinding hard resin.

(*d*) *Mechanical Agitator*.[4]

(*e*) *Temperature Regulator*, suitable means of maintaining temperatures at 25 ± 0.5 C (77 ± 0.9 F).

(*f*) *Viscosimeters* of any approved type agreed upon by the buyer and the seller giving accurate results in centistokes or results which can be converted to centistokes.

NOTE.—Capillary viscometers described in the Tentative Method of Test for Kinematic Viscosity (ASTM Designation: D 445),[3] or No. 4 Ford cup as described in the Tentative Method of Test for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup (ASTM Designation: D 1200),[5] are satisfactory.

Reagents

4. (*a*) *Resin* as agreed upon by the buyer and the seller.

(*b*) *Test Hydrocarbon Solvents.*

(*c*) *Control Solvent* as agreed upon by the buyer and the seller.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Published as information, June, 1953.

[3] 1952 Book of ASTM Standards, Part 5.

[4] Mechanical shakers that have been found to be satisfactory are either motor driven, reciprocating type shaking machines (A. H. Thomas Co., No. 8916) or Harbil Paint Mixers, $\frac{1}{16}$ to 1-gal capacity.

[5] 1952 Book of ASTM Standards Part 4.

Procedure

5. (*a*) *Hard Resins.*—Into a clean, dry, 16-oz narrow mouth, glass bottle, weigh the appropriate quantity of freshly ground lump resin to the nearest 0.05 g (Note 1). Add the necessary volume of control hydrocarbon solvent. The weight of resin and the volume of solvent shall be agreed upon by the buyer and the seller and shall be in such proportion that the resultant dispersion will have a viscosity of 100 to 500 centistokes. The total volume of the dispersion shall be at least 300 ml. Prepare similar mixes with equivalent amounts of resin and the test hydrocarbon solvent. Cork the bottles tightly and disperse the resin in the solvents by mixing at room temperature with a mechanical agitator until dispersion is complete (Note 2).

NOTE 1.—Since many resins are subject to oxidation in storage, only large lumps should be selected for grinding in order to minimize the effects of oxidation. The resin should be ground sufficiently fine to assure uniform dispersion of the material, but should not be ground to the consistency of a "flour" due to difficulty in handling in that state.

NOTE 2.—The test should be discontinued if the dispersion is not perfectly clear, since incomplete compatibility of resin and solvent is indicated.

(*b*) *Liquid Resins and Resin Solutions.*—Into a clean, dry, 16-oz wide mouth, glass bottle, weigh the appropriate quantity of liquid resin or resin solution to the nearest 0.05 g. If the resin or resin solution is very viscous, mild heating to a maximum temperature of 50 C will facilitate transfer to the bottle in which the dispersion is to be prepared, care being taken that solvents in a resin solution are not allowed to evaporate. Add the necessary volume of control hydrocarbon solvent. The weight of resin and volume of solvent shall be mutually agreed upon by the buyer and the seller and shall be in such proportion that the resultant dispersion will have a viscosity of 100 to 500 centistokes. The total volume of the solution shall be at least 300 ml. Prepare similar mixes with equivalent amounts of liquid resin or resin solution and test hydrocarbon solvents. Cap the bottles tightly and disperse the liquid resin or resin solution in the solvents by mixing at room temperature with a mechanical agitator for at least 15 min (Note 3).

If there is any lint or dirt in the dispersion after mixing is complete, the bottle should be centrifuged before viscosity determinations are made. In order to obtain reliable data, duplicate mixes should be prepared for each test. Determine the viscosity of each mix in centistokes at 25 C, using suitable temperature control, by means of any approved apparatus agreed upon by the buyer and the seller.

NOTE 3.—When solution is complete, bottles shall be allowed to stand at room temperature to eliminate air bubbles but viscosity shall be determined as soon as possible after bubbles have cleared.

Calculation

6. Calculate the viscosity reduction power, VRP, as follows:

$$\text{VRP} = \frac{\text{viscosity of control solvent dispersion in centistokes}}{\text{viscosity of test solvent dispersion in centistokes}} \times 100$$

When VRP is less than 100, the test hydrocarbon solvent has a lower VRP than the control. When VRP is greater than 100, the test solvent has a higher VRP than the control.

Precision

7. Results obtained in any one laboratory should not deviate from the average of two or more laboratories by more than ±15 per cent.

NOTE.—Limits of accuracy that may be expected with various types of viscosity equipment are found in the Standard Methods of Testing Varnishes (ASTM Designation: D 154),[5] Section 8(*b*) and (*c*).

APPENDIX II

PROPOSED METHOD OF TEST FOR FLASH POINT OF VOLATILE FLAMMABLE MATERIALS BY TAG OPEN-CUP APPARATUS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method describes a test procedure for the determination of open-cup flash points of volatile flammable materials having flash points below 175 F.

(*b*) This method, when applied to paints and resin solutions which tend to skin over or which are very viscous, gives less reproducible results than when applied to solvents.

NOTE 1.—A value in excess of 80 F by this method has been established by the U. S. Interstate Commerce Commission as the minimum value for classifying liquids as nonflammable. Materials having a flash point at or below that value must be identified with an ICC Red Label for interstate shipment by common carrier within the USA.[3]

Outline of Method

2. The sample is placed in the cup of a Tag Open Tester, and heated at a slow but constant rate. A small test flame is passed at a uniform rate across the cup at specified intervals. The flash point is taken as the lowest temperature at which application of the test flame causes the vapor at the surface of the liquid to flash, that is ignite but not continue to burn.

Apparatus

3. (*a*) *Flash Tester.*—Tag Open-Cup Tester (Fig. 1) as described in detail in the Supplement.

(*b*) *Shield.*—As described in detail in the Supplement.

(*c*) *Thermometer.*—For flash points above 40 F, use the ASTM Tag Closed Tester Thermometer, range of +20 to +230 F, in 1 F divisions, and conforming to thermometer 9F of ASTM Standard E 1.[4] For flash points below 40 F, use ASTM Tag Closed Tester, Low Range, thermometer 57F.

NOTE 2.—The original Tag Open Cup Thermometer is a permissible alternate. It is calibrated to −20 F.

Procedure

4. (*a*) Place the tester on a solid table free of vibration, in a location free of perceptible draft, and in a dim light.

(*b*) Run water, brine, or water-glycol solution into the bath to a predetermined level, which will fill the bath to

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

This proposed method is based on the Manufacturing Chemists' Association Draft for Flash Point Determination of Liquids for Classification under ICC Regulations by Tagliabue Open Cup.

[2] Published as information June, 1952; Revised, June, 1953.

[3] For details of ICC shipping regulations, see *Tariff 8*, issued March 15, 1951, by H. A. Campbell, agent, 30 Vesey St., New York 7, N. Y., publishing Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles by Land and Water in Rail Service and by Motor Vehicle (highway and water) including Specifications for Shipping Containers. (Regulations for transportation in rail express and rail baggage services are also included therein for information.)

[4] 1953 Supplements to Book of ASTM Standards, Parts 3 and 5.

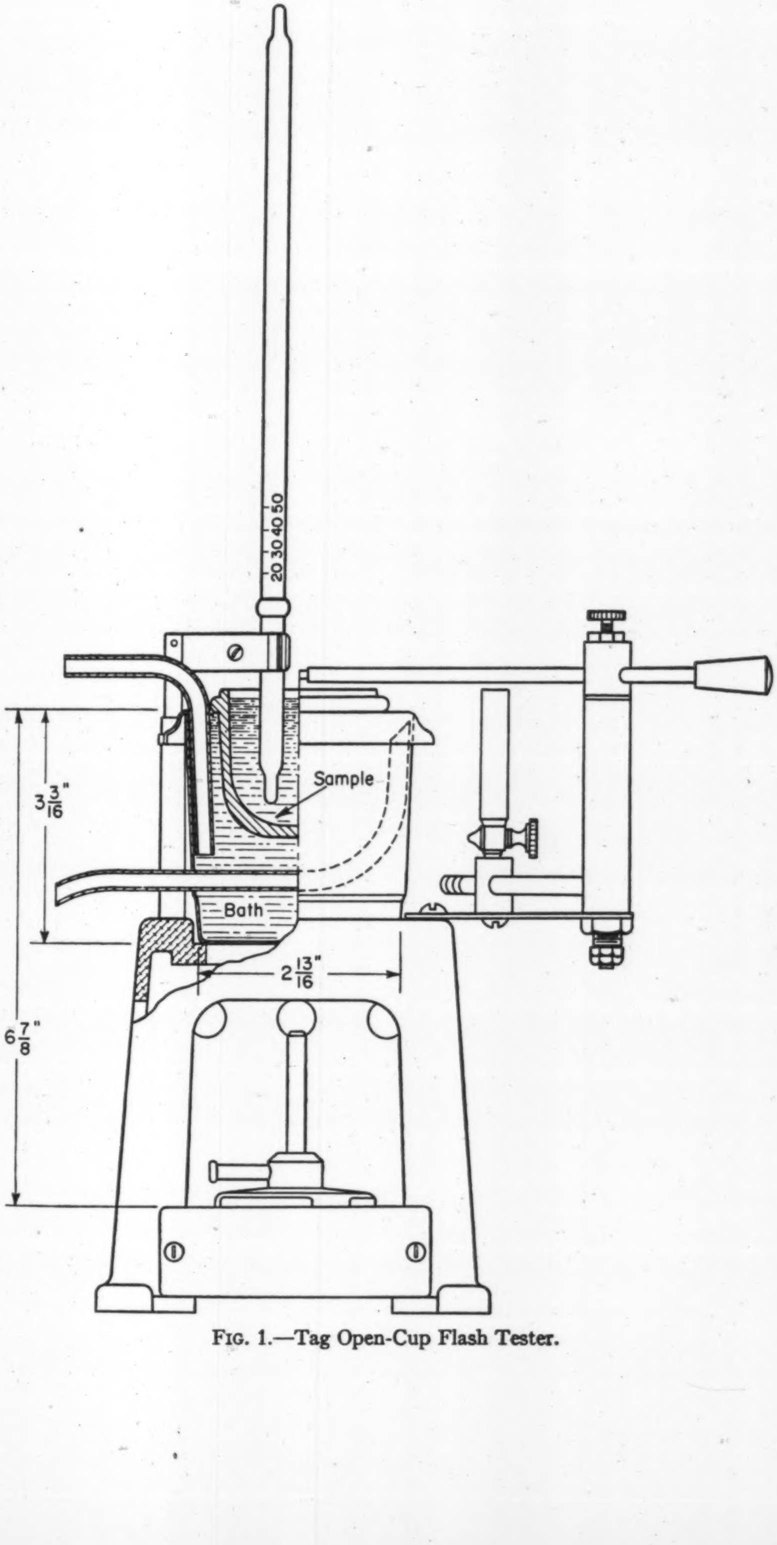
20 30 40 50
Sample
Bath
$3\frac{3}{16}$"
$6\frac{7}{8}$"
$2\frac{13}{16}$"

FIG. 1.—Tag Open-Cup Flash Tester.

$\frac{1}{8}$ in. below the top when the cup is in place. An overflow is permissible for water level control.

(*c*) Firmly support the thermometer vertically halfway between the center and edge of the cup on a diameter at right angles to the guide wire, or on a diameter passing through the center of the cup and the pivot of the taper. Place so that the bottom of the bulb is $\frac{1}{4}$ in. from the inner bottom surface of the cup.

NOTE 3.—If the old Tagliabue thermometer is used, immerse to well cover the mercury bulb, but not the wide body of the thermometer.

(*d*) Fill the glass cup with the sample liquid to a depth just $\frac{1}{8}$ in. below the edge, as determined by the leveling device.

(*e*) Place the guide wire or swivel device in position, and set the draft shield around the tester so that the sides form right angles with each other, and the tester is well toward the back of the shield.

(*f*) If a guide wire is used, the taper, when passed should rest lightly on the wire, with the end of the jet burner just clear of the edge of the guide wire. If the swivel type holder is used, the horizontal and vertical positions of the jet are so adjusted that the jet passes on the circumference of a circle, having a radius of at least 6 in., across the center of the cup at right angles to the diameter passing through the thermometer, and in a plane $\frac{1}{8}$ in. above the upper edge of the cup. The taper should be kept in the "off" position, except when the flame is applied.

(*g*) Light the ignition flame and adjust it to form a flame $\frac{3}{32}$ to $\frac{5}{32}$ in. in diameter.

(*h*) Place the heater flame under the center of the bath, and adjust it so that the temperature of the sample increases at a rate of 2 ± 0.5 F per min.

NOTE 4.—With viscous materials, this rate of heating cannot always be obtained.

Initial Test

5. Determine an approximate flash point by passing the taper flame across the sample at intervals of 2 F. The time required to pass the ignition flame across the surface of the sample should be about 1 sec.

NOTE 5.—Remove bubbles from the surface, of the sample liquid before starting a determination.

NOTE 6.—When determining the flash-point of viscous liquids and those liquids which tend to form a film of polymer, etc., on the surface, the surface film should be disturbed mechanically each time before the taper flame is passed.

Repeat Test

6. Repeat the procedure by cooling a *fresh* portion of the sample, the glass cup, the bath solution, and the thermometer at least 20 F below the approximate flash-point. Resume heating, and pass the taper flame across the sample at two intervals of 5 F and then at intervals of 2 F until the flash-point occurs.

Precision

7. For hydrocarbon solvents having flash points between 70 F and 110 F, the repeatability is ±2 F and the reproducibility ±5 F.

SUPPLEMENT

APPARATUS

A1. The Tag Open-Cup Tester is illustrated in Fig. 1. It consists of the following parts, which must conform to the dimensions shown, and have the additional characteristics as noted:

(*a*) *Copper Bath,* preferably equipped with a constant level overflow so placed as to maintain the bath liquid level $\frac{1}{8}$ in. below the rim of the glass cup.

(*b*) *Thermometer Holder.*—Support firmly with ring stand and clamp.

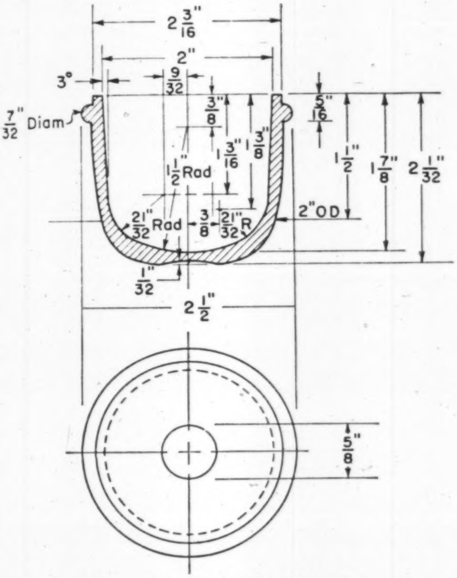

FIG. 2.—Glass Test Cup.

(*c*) *Glass Test Cup* (Fig. 2), of molded clear glass, annealed, heat-resistant, and free from surface defects.

(*d*) *Leveling Device,* or guide, for proper adjustment of the liquid level in the cup (Fig. 3). This shall be made of No. 18-gage polished aluminum, with a projection for adjusting the liquid level when the sample is added to exactly $\frac{1}{8}$ in. below the level of the edge or rim of the cup.

(*e*) *"Micro," or Small Gas Burner* of suitable dimensions for heating the bath. A screw clamp may be used to help regulate the gas. A small electric heater may be used.

(*f*) *Ignition Taper,* which is a small, straight, blow-pipe type gas burner. The test flame torch prescribed in the Standard Method of Test for Flash and Fire Points by Means of Open Cup (ASTM Designation: D 92)[5] is satisfactory.

(*g*) Alternate methods for maintaining the ignition taper in a fixed horizontal plane above the liquid may be used, as follows:

(*1*) *Guide Wire,* $\frac{3}{32}$ in. in diameter and $3\frac{1}{2}$ in. in length, with a right angle bend $\frac{1}{2}$ in. from each end. This wire is placed snugly in holes drilled in the rim of the bath so that the guide wire is $\frac{5}{8}$ in. from the center of the cup and resting on the rim of the cup.

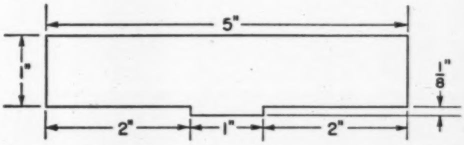

FIG. 3.—Leveling Device for Adjusting Liquid Level in Test Cup.

NOTE 5.—Two guide wires located $\frac{5}{8}$ and $\frac{7}{8}$ in., respectively, from the center of the cup and parallel to each other is a permissible alternate.

(*2*) *Swivel-Type Taper Holder,* such as is used in ASTM Method D 92. The height and position of the taper is fixed by adjusting the holder on a suitable ring-stand support adjacent to the flash cup.

(*h*) *Draft Shield,* consisting of two rectangular sheets of noncombustible material, 24 by 28 in., are fastened together along the 28-in. side, preferably by hinges. A triangular sheet, 24 by 24 by 34 in., is fastened by hinges to one of the lateral sheets (to form a top when shield is open). The interior of the draft shield shall be painted a flat black.

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[5] 1952 Supplement to Book of ASTM Standards, Parts 3 and 5.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS*

The following recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants have been approved by Committee D-2, in accordance with the regulations of the Society, and are presented herewith.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

Committee D-2 recommends that the following seven proposed methods be published as information only, as appended to this report,[1] or as otherwise indicated:

Appendix I. Functional Life of Ball Bearing Greases.
Appendix II. Chlorine in Lubricating Oils (Sodium Alcoholate Volumetric Method).
Appendix III. Method of Test for Sodium in Residual Fuel Oil—Flame Photometric Method.
Appendix IV. Recommended Practices for Applying Precision Data Given in ASTM Methods of Test for Petroleum Products and Lubricants.
Appendix V. Weathering Test of Liquefied Petroleum Gas (Based on NGAA Method).
Appendix VI. Proposed Methods for Analysis of Graphite.
Appendix VII. Test for Water in Petroleum Products and Bituminous Products (Published as information in 1952. See Appendix V, 1952 Report of Committee D-2).[2] The apparatus shown in Figs. 1 and 2 is to be revised as shown in the accompanying Figs. 1 and 2.

NEW TENTATIVES

Committee D-2 recommends that the following methods of test be accepted for publication as tentative:[3]

Tentative Methods of Test for:

Effect of Grease on Copper.
Leakage Tendencies of Automotive Wheel Bearing Grease.
Water Washout Characteristics of Lubricating Greases.
Sampling Liquefied Petroleum Gas. (This method has been removed from ASTM Method D 270 - 52 T, Sampling Petroleum and Petroleum Products.) This recommendation is submitted jointly with Committee D-3 on Gaseous Fuels.
Sulfur in Petroleum Products and Liquefied Petroleum Gases by the CO_2-O_2 Lamp Method. (This method is an enlargement of Appendix VI, 1949 Report of Committee D-2.)
Lead in New and Used Greases.
Unsaturated Light Hydrocarbons (Silver—Mercuric Nitrate Absorption).
Vapor Pressure of Liquefied Petroleum Gas.
Polarographic Determination of Tetraethyllead in Gasoline. (See Appendix VI, 1952 Report of Committee D-2.)[4] This method is to be revised as follows:

Title.—Add a footnote reference after the Title; the footnote to read:

"Research Division III on Elemental Analysis plans to investigate cooperatively another polarographic method based on an instrument developed by Offut and Sorg which is described in *Analytical Chemistry*, Vol. 22, p. 1234 (1950). The instrument indicates TEL content directly when fixed potentials are applied by means of push buttons across the electrolysis cell. Following the investigation, consideration will be given to the inclusion of such a procedure in this tentative method."

Add a new Note 1 to read as follows, renumbering subsequent notes and references thereto in the text accordingly:

"NOTE 1.—Another method, Method D 526, Test for Tetraethyllead in Gasoline, is also available for this purpose. These two methods give equivalent results within their stated precision limits."

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] See pp. 364, 369, 372, 379, 384, and 387.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 358 (1952).

[3] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 5.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 365 (1952).

Precision.—Add a Section 8 on Precision to read:

"8. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability."

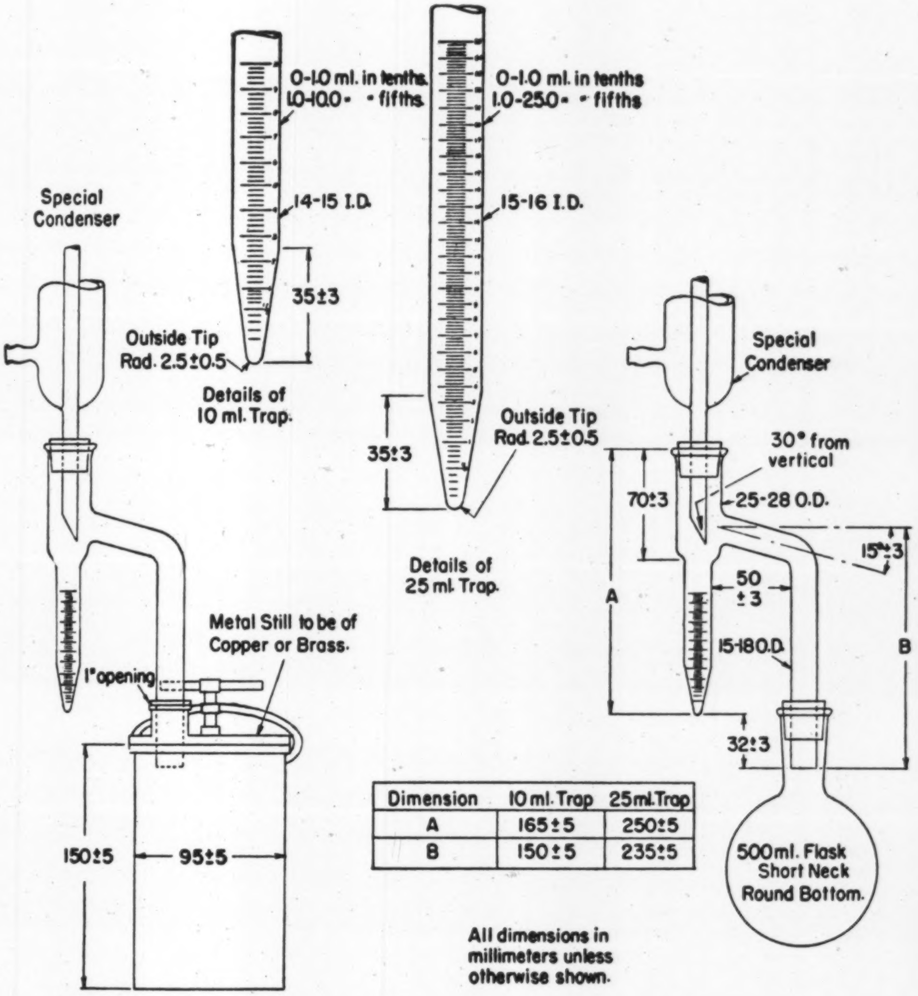
(*a*) Duplicate results obtained by the same operator should be considered suspect if they differ from each other by more than the following amounts:

Repeatability.... 0.03 + 0.022 (TEL content in ml per U S gal).

(*b*) The results submitted by each of two laboratories should be considered suspect if they differ by more than the following amounts:

Reproducibility.... 0.06 + 0.027 (TEL content in ml per U S gal).

Table I.—Delete.


0-1.0 ml. in tenths
1.0-10.0 " fifths
14-15 I.D.
35±3
Outside Tip
Rad. 2.5±0.5
Details of
10 ml. Trap.
0-1.0 ml. in tenths
1.0-25.0 " fifths
15-16 I.D.
35±3
Outside Tip
Rad. 2.5±0.5
Details of
25 ml. Trap.
Special
Condenser
1" opening
Metal Still to be of
Copper or Brass.
150±5
95±5
Special
Condenser
30° from
vertical
70±3
25-28 O.D.
15°±3
50
±3
A
15-18 O.D.
B
32±3
500 ml. Flask
Short Neck
Round Bottom.
All dimensions in
millimeters unless
otherwise shown.

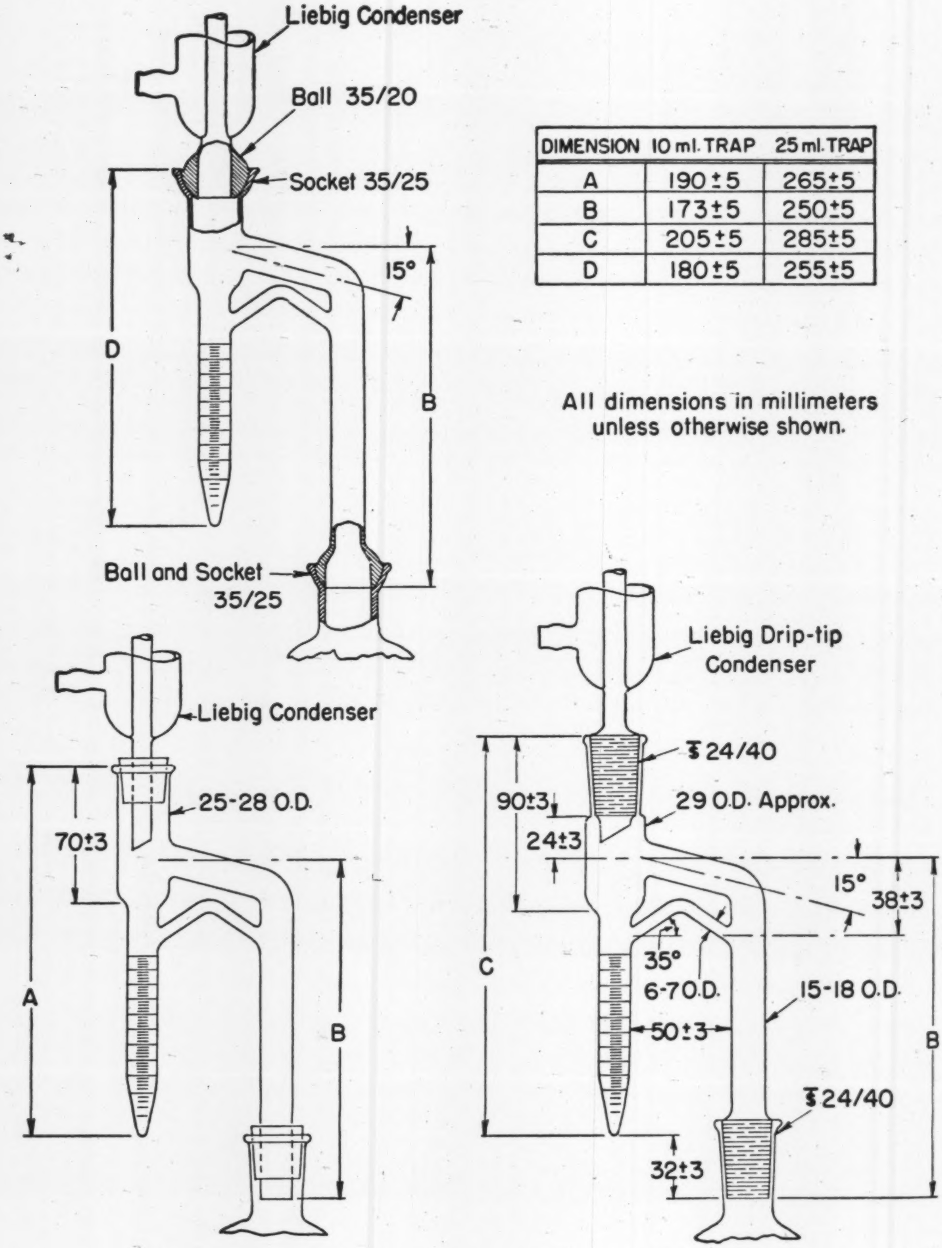
Dimension	10 ml. Trap	25 ml. Trap
A	165±5	250±5
B	150±5	235±5

FIG. 1.—Apparatus for Determining Water in Petroleum and Bituminous Products.

REVISION OF TENTATIVES

Committee D-2 recommends revision of the following 12 tentatives, as indicated:

D 270 – 52 T,[5] Sampling Petroleum and


Liebig Condenser
Ball 35/20
Socket 35/25
15°
D
B
Ball and Socket 35/25
Liebig Condenser
25-28 O.D.
70±3
A
B
Liebig Drip-tip Condenser
₮ 24/40
90±3
29 O.D. Approx.
24±3
15°
38±3
C
35°
6-7 O.D.
15-18 O.D.
50±3
B
₮ 24/40
32±3

DIMENSION	10 ml. TRAP	25 ml. TRAP
A	190±5	265±5
B	173±5	250±5
C	205±5	285±5
D	180±5	255±5

All dimensions in millimeters unless otherwise shown.

FIG. 2.—Construction Details of Condensers for Water Determination.

Petroleum Products, revise as follows:

Section 1.—Change the scope to read:

"1. This method describes the standard procedures for obtaining representative samples of stocks of shipment of petroleum and its products, except butanes, propanes, and other gases.

"NOTE 1.—The procedure for sampling liquefied petroleum gases is described in ASTM Method D 1265, and for sampling electrical insulating oils in ASTM Method D 923."

Table I.—In the sub-table on sampling for specific tests, delete the third line from bottom which reads "D 923 Electrical Insulating Oils . . . . 57."

Table II.—Delete "Sampling Liquefied Petroleum Gases" and corresponding references under each column of the main table.

Section 4(d).—Delete.

Section 8(a).—Delete "(Except Liquefied Petroleum Gases)" from paragraph heading.

Section 8(c).—Delete ". . . such as electrical insulating oils."

Section 8(d).—Delete "See Section 49 for Liquefied Petroleum Gases."

Section 9.—Delete the last sentence.

Sections 43 to 51.—Delete the entire procedure for sampling liquefied petroleum gases and also Fig. 7. Renumber other sections accordingly.

Section 52.—Delete "D 923 Electrical Insulating Oils . . . .57" in reference table.

Section 57.—Delete this entire procedure for sampling *electrical insulating oils.*

D 445 - 52 T,[5] Kinematic Viscosity. The method has been revised as appended hereto,[6] and a number of appendices describing the various viscometers have been included.

D 526 - 48 T,[5] Tetraethyllead in Gasoline, revise as follows:

Add a new Note 1 to read as follows, renumbering subsequent notes and references thereto in the text accordingly:

"NOTE 1.—Another method, Method D 1269, Test for Polarographic Determination of Tetraethyllead in Gasoline, is also available for this purpose. These two methods give equivalent results within their stated precision limits."

Section 4(h).—Include the following as a new Section 4(*h*), relettering the present paragraphs accordingly:

"(*h*) *Potassium Chlorate—Nitric Acid Solution.*—Dissolve 78 g of potassium chlorate in 550 ml of concentrated nitric acid."

Section 5(b).—Change to read:

"(*b*) Evaporate the aqueous extract to dryness (Note 4). Add 3 ml of concentrated nitric acid to the residue and heat to oxidize any organic material present (Note 5). Repeat the nitric acid treatment. If a white residue is not obtained after two additions of nitric acid, oxidize the remaining organic matter with the potassium chlorate-nitric acid mixture as described in Note 5. Add 4 ml of dilute nitric acid mixture and 25 ml of distilled water; heat until all the lead salt is in solution."

Note 4.—Continue Note 3 without change and add a new Note 5 to read:

"NOTE 5.—If the residue flashes on being heated with nitric acid, the sample should be discarded and the acid extraction repeated on another sample of the gasoline. Then evaporate the extract until crystallization commences, but not to complete dryness. Add 10 ml of potassium chlorate-nitric acid mixture, cover the beaker with a watch glass and evaporate the mixture almost to dryness. Repeat this treatment, if necessary, to obtain a white residue."

Section 6.—Revise to read:

"6. *Calculation.*—Calculate the concentration of tetraethyllead as follows:

$$\text{Tetraethyllead} = K \times \frac{G}{V}$$

where:

G = grams of lead chromate,

V = milliliters of sample (50 or 100 ml), and

K = multiplier varying with sample temperature as given in Table I or Table II."

Table I.—Add a new Table I as follows:

TABLE I.—VALUES OF K.

Sample Temperature, deg Fahr	Reporting Units		
	ml per U S gal at 60 F	ml per Imperial gal at 60 F	ml per liter at 60 F
50	2274	2731	600.5
55	2282	2740	603.0
60	2290	2750	605.0
65	2298	2760	607.0
70	2306	2770	609.0
75	2314	2779	611.5
80	2322	2789	613.5
85	2331	2800	616.0
90	2340	2810	618.0
95	2348	2820	620.0

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[5] 1952 Book of ASTM Standards, Part 5.

[6] The revised method was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 5.

Note 6.—Add new Note 6 to read:

"NOTE 6.—The multiplier *K* is obtained by using 1.65 as the density of tetraethyllead at 60 F, 0.9981 as an empirical factor for converting lead chromate to TEL, 3785.3 and 4546.1 as the number of milliliters of U S and Imperial gallons, respectively, and Group 4 factors of Table 7 or 25, Abridged Volume Correction Tables of ASTM D 1250 and IP 200 Petroleum Measurement Tables."

Table II—Add a new Table II as follows:

TABLE II.—VALUES OF *K*.

Sample Temperature, deg Cent	Reporting Unit, ml per liter at 15 C
10	601.1
15	604.9
20	608.8
25	612.7
30	616.7
35	620.7

Note 7.—Add new Note 7 to read:

"NOTE 7.—The multiplier *K* is obtained by using 1.65 as the density of tetraethyllead at 15 C, 0.9981 as an empirical factor for converting lead chromate to TEL, and Group 4 factors of Table 55, Abridged Volume Correction Tables, of ASTM D 1250 and IP 200, Petroleum Measurement Tables."

Section 7.—Revise to read:

"7. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability)."

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Repeatability.... 0.03 + 0.022 (TEL content in ml per US gal).

(*b*) The results submitted by each of two laboratories should be considered suspect if they differ by more than the following amounts:

Reproducibility.... 0.06 + 0.027 (TEL content in ml per US gal).

D 611 - 51 T,[5] Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents, revise as follows:

Note 1.—Delete, and renumber subsequent Notes.

Section 4(a).—Change to read:

"4. (*a*) *Test Tube or Thin-Film Apparatus,* made of heat-resistant glass and conforming to the dimensions prescribed in the Appendix."

Table I.—Replace with the following:

TABLE I.—REQUIREMENTS FOR *n*-HEPTANE.

Octane Number, ASTM Motor	−0.2 to +0.2
Density at 20 C, g per ml	0.68375 ± 0.00015
Refractive Index, n_D^{20C}	1.38775 ± 0.00015
Freezing Point[a], deg Cent	−90.72 min
Distillation[b]:	
Temperature of 50 per cent recovery, deg Cent	98.43 ± 0.05
Temperature increases from 20 to 80 per cent recovery, deg Cent	0.20 max

Footnotes "a" and "b".—Continue without change.

Section 7.—Change to read:

"7. Two methods, to be used as applicable, are covered as follows:

Method 1, described in detail in the Appendix, is applicable to clear, light-colored samples or to samples not darker than No. 8 ASTM color, as determined by ASTM Method D 155, Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter.

Method 2, described in detail in the Appendix, is applicable to light-colored samples, to moderately dark samples, and very dark samples."

Note 4.—Add Note 4 to read:

"NOTE 4.—The U-tube equipment for the determination of aniline point, which appeared as Method B in the Appendix to ASTM Method D 611 - 52 T, is an acceptable alternate, but has been dropped since Methods 1 and 2 together are adequate for all samples covered by the scope of this method."

Appendix.—Revise as follows:

Method A.—Retitle as Method 1.

Section A2, Paragraph (b).—Give the number 5 to the unnumbered note and to the reference to the Note in this section. Change reference from Note 4 to Note 3.

Method B.—Delete.

Figs. 2 and 3.—Delete.

Method C.—Retitle as Method 2.

Section A5 and A6.—Renumber as A3 and A4.

Figs. 4 and 5.—Renumber as Figs. 2 and 3, respectively; also renumber references in text of present Sections A5 and A6 accordingly.

Section A6, Paragraph (a).—Change Note 4 to Note 3.

Section A6, Paragraph (b).—Give the number 6 to the Note and to the reference to this Note in the Section.

Footnote 7.—Change to Footnote 6 to agree with reference in the test.

D 665 - 52 T,[5] Rust-Preventing Characteristics of Steam Turbine Oil in

the Presence of Water, revise as follows:

Section 4 (*a*)—Change last sentence to read: "The steel specimen, either new or from a previous test (Note 4), shall be prepared as described in Paragraphs (*b*) and (*c*)."

Add a new Note 4 to read:

"NOTE 4.—When making a check test, the steel specimen that showed rust should not be re-used. Specimens that repeatedly show rust in tests of various oils may be imperfect. Such specimens should be used with oils known to pass the test. If rusting occurs in repeat tests, these specimens should be discarded."

Renumber subsequent Notes 4, 5, and 6, and references thereto, accordingly.

Section 7.—Revise this section and Note 7 to read:

"7. *Report.*—(*a*) Since the test may be performed either by Procedure A or Procedure B, and since it may be conducted for various periods of time (Note 7), reports of results should indicate the procedure followed, for example, Rusting Test, 24 hr with distilled water; or, Rusting Test, 48 hr with synthetic sea water.

(*b*) All inspections at the end of the test to determine the condition of specimens shall be performed without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 60 foot candles. Within the meaning of this method, a rusted specimen is one on which any rust spot or streak is visible by the above inspection procedure.

(*c*) In order to report an oil as passing or failing, the test must be conducted in duplicate. An oil shall be reported as passing the test if both specimens are rust-free at the end of the test period. An oil shall be reported as failing the test if both specimens are rusted at the end of the test period (Note 8). If one specimen is rusted while the other is free of rust, tests on two additional specimens shall be made (Note 4). If either of these latter specimens shows rusting, the oil shall be reported as not passing the test. If neither of these latter specimens shows rusting, the oil shall be reported as passing the test."

NOTE 8.—An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended:

Light rusting.—Rusting confined to not more than six spots, each of which is 1 mm or less, in diameter.

Moderate rusting.—Rusting in excess of the above but confined to less than 5 per cent of the surface of the specimen.

Severe rusting.—Rusting covering more than 5 per cent of the surface of the specimen."

D 875 - 46 T,[5] Olefins and Aromatics in Petroleum Distillates. This Method has been rewritten as appended hereto.[6]

D 910 - 52 T,[5] Aviation Gasolines, revise as follows:

Section 2.—Revise to read:

"2. *Grades.*—Five grades of aviation gasoline are provided, known as:

Grade 80–87
Grade 91–98
Grade 100–130
Grade 108–135
Grade 115–145"

Table I.—Add a new column of requirements for Grade 108–135 to read:

	Grade 108–135
Knock value, min, octane number, lean rating	*iso*octane plus 0.22 ml. tetraethyllead per gallon
Knock value, min, octane number, rich rating	*iso*octane plus 1.68 ml. of tetraethyllead per gallon
Color	brown
Dye Content:	
Permissible blue dye, max, mg per gal	3.12
Permissible yellow dye, max, mg per gal	none
Permissible red dye, max, mg per gal	2.7
Permissible orange dye, max, mg per gal	6.0
Tetraethyllead, max, ml per gal	3.0
Net heat of combustion, min, Btu per lb	18 800

Also in Table I change the requirements for permissible maximum blue as follows:

	Grade 80/87	Grade 91/98	Grade 100/130	Grade 115/145
"Dye Content:				
Permissible blue dye[d] max, mg per gal	0.5	5.7	4.7	4.7"

In the case of the new dye, permissible orange, as specified above for Grade 108–135, in Table I add "none" for this item for Grades 80–87, 91–98, 100–130, 115–145.

Following footnote g, add the parenthetical phrase "(Color Index No. 24)."

Reletter all subsequent footnotes and references to them appearing in Table I.

Section 9(c).—Change to read as follows including Notes 1 and 2 below:

"(*c*) *Color.*—Color comparison may be made by any suitable apparatus or by visual examination, using identical transparent containers for the aviation gasoline sample and for the appropriate maximum and minimum color standards (Notes 1 and 2). Color standards which have been exposed to light more than 24 hrs shall not be used for this test."

"NOTE 1.—One-gallon maximum and minimum color standard samples for Grades 80–87, 91–98, 100–130, and 115–145 may be obtained upon request to the Commanding General, Air Materiel Command, Wright Field, Dayton, Ohio. Requests for such samples shall include the following information: identification of color standard desired (color and whether maximum- or minimum-intensity), number of containers desired, and consignee address."

"NOTE 2.—One-gallon maximum and minimum color standard samples for grade 108–135 may be obtained from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa., at the cost of one dollar per sample."

D 943 - 47 T,[5] Oxidation Characteristics of Inhibited Steam Turbine Oils, revise as follows:

Section 4(a).—Change to read:

"4(*a*). Catalyst of low metalloid steel wire[4], No. 16 Washburn and Moen gage (0.0625 in. diameter) and of electrolytic copper wire No. 14 American Wire gage (0.064 in. diameter)."

Footnote 4.—Revise to read:

"[4]ASTM Specifications A 129 - 50 T type "C" low metalloid steel wire is satisfactory. If this steel is not available, other equivalent steels may be used, provided they are found to be satisfactory in comparative tests using ASTM Method D 943, Test for Oxidation Characteristics of Inhibited Steam Turbine Oils."

D 974 - 52 T,[5] Neutralization Value (Acid and Base Numbers) by Color Indicator Titration, revise as follows:

Section 7(c).—Add the following new Note 7; renumbering present Note 7 as 8.

"NOTE 7.—The titration solvent usually contains weak acid impurities which react with the strongly basic components of the sample. To correct the strong-base number for the sample, it is thus necessary to determine an *acid number* blank upon the solvent."

Section 8.—Change to read:

"8. *Procedure for Strong Base Number.*—(*a*) If the titration solvent containing the dissolved sample assumes a green or greenish-brown color after the indicator is added (Section 7(*a*)), carry out the titration as described in Section 7(*b*), but use 0.1 N HCl and titrate until the green-brown color changes to orange.

(*b*) *Blank.* Make a blank titration as directed in Section 7(*c*), (Note 7).

Section 9(b).—Revise to read:

"(*b*) *Blank.*—Into a 250-ml Erlenmeyer flask, introduce 200 ml of the same boiling distilled water as used for the sample titration. Add 0.1 ml of methyl orange indicator solution. If the indicator color is yellow-orange, titrate with 0.1 *N* HCl to the same depth and shade of color obtained in the titration of the sample. If the indicator color is pink or red, titrate with 0.1 *N* KOH to the same end point as that used in the sample titration."

Section 10.—Revise to read:

"10. *Calculations.*—(*a*) Calculate the total acid number as follows:

Total Acid Number, mg of KOH per g

$$= \frac{(A - B)N \times 56.1}{W}$$

where:

A = milliliters of KOH solution required for titration of the sample (Section 7(*b*)),

B = milliliters of KOH solution required for titration of the blank (Section 7(*c*)),

N = normality of the KOH solution, and

W = grams of sample used.

(b) Calculate the strong-acid number as follows:

(*1*) If the blank titration is made with acid:

Strong-acid number, mg of KOH per g

$$= \frac{(CN + Dn) \times 56.1}{W}$$

where:

C = milliliters of KOH solution required to titrate the water extract (Section 9(*a*)),

D = milliliters of HCl solution required to titrate the blank solution (Section 9(*b*)),

N = normality of the KOH solution,

n = normality of the HCl solution, and

W = grams of sample used.

(*2*) If the blank titration is made with base:

Strong-acid number, mg of KOH per g

$$= \frac{(C - D)N \times 56.1}{W}$$

where:

C = milliliters of KOH solution required to titrate the water extract (Section 9(*a*)),

D = milliliters of KOH solution required to titrate the blank solution (Section 9(*b*)),

N = normality of the KOH solution, and

W = grams of sample used.

(c) Calculate the strong base number as follows:

Strong-base number, mg of KOH per g

$$= \frac{(En + FN) \times 56.1}{W}$$

where:

E = milliliters of HCl solution required for titration of the sample (Section 8),

F = milliliters of KOH required for titration of the acid number blank,

n = normality of the HCl solution,

N = normality of the KOH solution, and

W = grams of sample used."

D 975 – 52 T,[5] Classification of Diesel Fuel Oils, revise as follows:

Table I.—Make the following changes:

Delete the numerical value of 20 for the grade No. 1-D fuel in the column headed "Pour Point, deg. Fahr.," but retain footnote "*b*."

Delete footnote "*d*" from the present classification.

Add a letter "*d*" opposite "Cetane Number," and include a footnote "*d*" to read:

"[d] Where cetane number by the Cetane Method (ASTM Method D 613) is not available, Calculated Cetane Index, see Appendix II, may be used as an approximation. Where there is disagreement, the Cetane Method (ASTM Method D 613) shall be the referee method."

Appendix II.—Delete footnote 5; renumber subsequent footnotes.

D 1021 – 49 T,[5] Oxygen in Butadiene Vapors (Manganous Hydroxide Method), revise as follows:

Section 1.—Revise the scope to read:

"1. This method describes a procedure for the determination of low concentrations of oxygen (0.02 to 6.0 per cent) in the vapor above liquid butadiene when the sample does not contain such oxidizing or reducing gases as hydrogen sulfide, sulfur dioxide, and the oxides of nitrogen."

Section 3.—Add a new Paragraph (*c*) to read:

"(*c*) *Mechanical Agitator*, giving vigorous agitation, in which the liquid shall be thrown from side to side in the flask, breaking and reestablishing the liquid surface on each cycle."

Section 4(a)—Change to read:

"(*a*) *Manganous Chloride Solution* (50 g $MnCl_2$ per l).—Dissolve 78.6 g of $MnCl_2 \cdot 4H_2O$ in water and dilute to 1 liter."

Section 6(a)—Change to read:

"6. (*a*) Wash the apparatus thoroughly. After drying the outside and inside of the flask, tubes and calibrated cylindrical funnel, connect to a vacuum line at a pressure of 1 mm or less to complete drying."

Section 6(c)—Change sentence one to read as follows: "Place 75 ml of $MnCl_2$ solution in the calibrated tube of the separatory funnel." Change sentence 4 to read:

"Add 15 ml of NaOH solution....flask .... flask; shake the flask vigorously in the mechanical agitator continuously for 10 min."

Section 8.—Revise to read:

8. *Precision.*—Results should not differ from the mean by more than the following amounts:

Oxygen Content, per cent by volume	Repeatability, Same Operator and Apparatus	Reproducibility, Different Operators and Apparatus
0.1 to 1.0........	0.02	0.04
1.0 to 6.0........	0.10	0.20"

D 721 – 51 T, Oil Content of Paraffin Waxes,[5] replace this method by the Method of Test for Oil Content of Petroleum Waxes published as information in Appendix IX of the 1952 Report of Committee D-2,[7] revising Section 8 to read as follows:

"*Precision.*—8. The following data should be used for judging the acceptability of results (95 per cent probability).

(*a*) Duplicate results by the same operator should be suspect if they differ by more than the following amounts:

Oil Content, per cent	Repeatability
Under 2............................	0.1 per cent
2 and over..........................	5 per cent of mean

(*b*) The results submitted by each of two laboratories should be suspect if they differ by more than the following amounts:

Oil Content, per cent	Reproducibility
Under 2.........................	0.2 per cent
2 and over......................	7.5 per cent of mean or 0.2 per cent (absolute) whichever is greater"

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Committee D-2 recommends that the

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[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 380 (1952).

Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (D 156 - 49) be revised and reverted to tentative. This method has been extensively revised as appended hereto.[6]

ADOPTION OF TENTATIVES AS STANDARD

Committee D-2 has reviewed the following five tentatives which have stood at least one year without revision, and which represent best present-day practices, and recommends that they be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

D 1024 - 49 T. Butadiene Dimer in Polymerization-Grade Butadiene,

D 1088 - 51 T. Boiling Point Range of Polymerization Grade Butadiene.

D 1089 - 50 T. Carbonyl Content of Butadiene, revised as follows:

Section 3(b).—Change to read:

(*b*) *Thermometer.*—For observing temperatures below −45 C, the Low Cloud and Pour Thermometer, range −80 to 20 C (−112 to 70 F), conforming to the requirements for thermometer No. 6 C (6 F), in the ASTM Specifications E 1 is satisfactory."

D 1094 - 50 T. Water Tolerance of Aircraft Fuels, and

D 1250 - 52 T. ASTM-IP Petroleum Measurement Tables.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee D-2 recommends for immediate adoption revisions of ten standards as indicated below, and requests a nine-tenths affirmative vote at the Annual Meeting so that these revisions may be referred to Society letter ballot:

D 86 - 52.[5] Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products. Revise as follows:

Section 3(c)—Change to read:

"(*c*) A 100-ml sample shall be measured in the 100-ml graduated cylinder at 55 to 65 F (12.8 to 18.3 C) and transferred directly to the distillation flask which has been cooled at 55 to 65 F (12.8 to 18.3 C) for gasolines higher than 9.5 lb. Reid vapor pressure. The flask may be at room temperature for other products. None of the liquid shall be permitted to flow into the vapor tube."

Section 4(c)—Insert a reference to "Note 4" after the second sentence, and include the following Note 4 at the end of this paragraph:

"NOTE 4.—When distilling certain naphthas manufactured for specific purposes, such as for use in paints and varnishes, the temperature indicated by the distillation thermometer when the last drop evaporates from the lowest point in the flask shall be recorded as the ***dry point***. Any liquid hanging to the side of the flask or the thermometer should be disregarded."

Section 4(e).—Revise to read: "(*e*) The total volume of the distillate collected in the receiving graduate shall be observed at the conclusion of the distillation, and thereafter at not less than 2-min intervals, until successive observations agree. This volume shall then be recorded as the ***recovery***."

Section 4 (h).—Insert reference to a Note 5, and include the following Note 5 at the end of this paragraph:

"NOTE 5.—Abnormal distillation losses, when caused by low barometric pressure, may be corrected by the equation given below to an approximation of the loss to be expected under normal pressure. The corrected losses shall not be used in the calculation of percentages evaporated.

$$\text{Corrected Loss} = AL + B$$

where:

L = observed distillation loss, and

A and B = empirical Constants, as given in Table II for various values of the observed barometric pressure."

Table II.—Include a new Table II, as follows:

TABLE II.—VALUES OF THE CONSTANTS "A" AND "B" USED IN OBTAINING CORRECTED DISTILLATION LOSS.

Observed Barometric Pressure, mm	*A*	*B*	Observed Barometric Pressure, mm	*A*	*B*
560........	0.231	0.384	660......	0.375	0.312
570........	0.240	0.380	670......	0.400	0.300
580........	0.250	0.375	680......	0.428	0.286
590........	0.261	0.369	690......	0.461	0.269
600........	0.273	0.363	700......	0.500	0.250
610........	0.286	0.357	710......	0.545	0.227
620........	0.300	0.350	720......	0.600	0.200
630........	0.316	0.342	730......	0.667	0.166
640........	0.333	0.333	740......	0.750	0.125
650........	0.353	0.323	750......	0.857	0.071
			760......	1.000	0.000

Section 4(i).—Change first sentence to read: "The sum of the volume collected in the cylinder at any specified temperature, including the initial boiling point, and the distillation loss may be recorded as the percentage evaporated at the temperature in question. Clear . . . recovered."

Section 6.—Change title of the section to read: "Correction of Temperature Points to Standard Atmospheric Pressure."

D 88 – 44.[5] Viscosity by Means of the Saybolt Viscosimeter.

Eliminate all reference to Bureau of Standards' master Saybolt oil tube by making the following revisions:

Section 2(a).—Change the last sentence to read as follows: "The time of flow shall be within ±1 per cent (Note 1) of the outflow time for American Petroleum Institute oil standards or National Bureau of Standards oil standards (Note 2)."

Note 1.—Delete all but the first paragraph.

Note 2.—Delete and renumber Note 3 as 2.

D 158 – 52.[5] Distillation of Gas Oil and Similar Distillate Fuel Oils. Revise as follows:

Section 2(a)—Change line 4 in the table of dimensions, length of vapor tube, to read:

	cm	in.	Permissible Variations	
			cm	in.
Length of vapor tube...............	10.0	3.94	±0.3	±0.12

D 216 – 52.[5] Distillation of Natural Gasoline, revise as follows:

Section 5(d)—Change to read:

"(*d*) The total volume of the distillate collected in the receiving graduate shall be observed at the conclusion of the distillation, and thereafter at not less than 2-min intervals, until successive observations agree. This volume shall then be recorded as the *recovery*."

Section 5(g)—Change first sentence to read: "The sum of the volume collected in the cylinder at any specified temperature, including the initial boiling point, and the distillation loss shall be recorded as the percentage evaporated at the temperature in question. Clear . . . *recovered*."

Section 5(f)—Insert reference to a "Note 2," and include the following Note 2 at the end of this paragraph:

"Note 2.—Abnormal distillation losses, when caused by low barometric pressure, may be corrected by the equation given below to an approximation of the loss to be expected under normal pressure. The corrected losses shall not be used in the calculation of percentages evaporated."

$$\text{Corrected Loss} = AL + B$$

where:

L = observed distillation loss,

A and B = empirical constants, as given in Table I, for various values of the observed barometric pressure.

Table I.—Include a new Table I, the text of which will be the same as the proposed new Table II in Method D 86 given earlier in this report.

Section 7.—Change title to read: "Correction of Temperature Points to Standard Atmospheric Pressure."

D 288 – 52,[5] Definition of Terms Relating to Petroleum, revise as follows:

Add a new definition for Stoddard Solvent to read:

"*Stoddard Solvent*.—A refined petroleum distillate with volatility, flash point, and other properties making it suitable for use as a dry cleaning solvent.

"Note.—Specifications are set forth in detail in the Standard Specifications for Stoddard Solvent (ASTM Designation: D 484)."

D 357 – 49,[5] Knock Characteristics of Motor Fuels by the Motor Method. The bouncing pin is to be eliminated from the method, and shall not be used after January 1, 1954. Revise the Method as follows:

Section 3.—In line 11 delete "a bouncing pin and knockmeter or by."

Section 4.—In line 7 delete "Either a bouncing pin[3] and knockmeter or."

Footnote 3.—Delete; renumber subsequent footnotes.

Table I.—Delete; renumber Table IA as Table I.

Section 6(n).—Delete "and bouncing pin."

Tables III and IV.—Delete "For Bouncing Pin or Detonation Meter" from the titles.

Sections 6(p) and 8.—Delete "IA."

Sections 9 and 10.—Delete; renumber subsequent sections.

Fig. 2.—Delete; renumber Fig. 3 as Fig. 2.

Section 12(a).—Delete lines 7 through 21; replace with: "If this match cannot be obtained under standard conditions, check the mechanical condition of the engine."

Section 12(b).—In sentence one delete the phrase: "provided no change has been made in the bouncing pin or detonation meter adjustment in the meantime." In sentence two delete "IA."

A list of other revisions necessary in the ASTM Manual of Engine Test Methods for Rating Fuels to eliminate the bouncing pin therefrom is appended hereto.[8]

D 446 – 39.[5] Conversion of Kinematic to Saybolt Universal Viscosity.

This standard has been revised as appended hereto[6] in order to bring the method in line with a new value for the viscosity of water (1.0038 cs at 20 C) to be adopted by the National Bureau of Standards on July 1, 1953. An extension of Table I is being prepared and will be available by June, 1953, in a separate ASTM publication.[9]

D 567 – 41.[5] Calculating Viscosity Index.

This revised standard as appended hereto[6] also will bring this method in line with a new value for the viscosity of water (1.0038 cs at 20 C), to be adopted by NBS on July 1, 1953. An extension of Table II is being prepared, and will be available by June, 1953, in a separate ASTM publication.[9]

D 666 – 44.[5] Conversion of Kinematic Viscosity to Saybolt Furol Viscosity.

This standard has also been revised as appended hereto[6] in order to bring this method in line with a new value for the viscosity of water (1.0038 cs at 20 C), to be adopted by NBS on July 1, 1953. An extension of Table I is being prepared, and will be available by June, 1953, in a separate ASTM publication.[9]

D 908 – 51.[5] Knock Characteristics of Motor Fuels by the Research Method.

The bouncing pin is to be eliminated from the method, and shall not be used after January 1, 1954. Revise the method as follows:

Section 3.—In line 20 delete "bouncing pin and knockmeter or by a."

Section 4.—In line 15 delete "Either a bouncing pin[3] and knockmeter or."

Footnote 3.—Delete; renumber subsequent footnotes.

Table V.—Delete; renumber Table VA as Table V.

Section 6(m).—In line 6 delete "and bouncing pin."

Section 6(o).—In line 9 delete "VA."

Section 8.—In line 6 delete "or VA."

Sections 9 and 10.—Delete; renumber subsequent sections.

Section 12(a).—Delete lines 7 through "test fuels" on line 22. Replace with: "If this match cannot be obtained under standard conditions, check the mechanical condition of the engine."

Section 12(b).—In sentence one delete the phase: "provided no change has been made in the bouncing pin or detonation meter adjustment in the meantime." In sentence two delete "or VA."

A list of other revisions necessary in the ASTM Manual of Engine Test Methods for Rating Fuels to eliminate the bouncing pin therefrom is appended hereto.[8]

WITHDRAWAL OF STANDARD

Committee D-2 recommends the withdrawal of the Standard Method of Test for Existent Gum in Gasoline (Air-Jet Evaporation Method) (D 381 – 50). The method is to be replaced by Method D 381 – 52 T, Test for Existent Gum in Fuels by Jet Evaporation.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-2 has reviewed the following 18 tentatives, which have stood for two or more years without revision, and, for good and sufficient reasons discussed within its subordinate groups, recommends that they be continued as tentatives without revision:

Tentative Specifications for:

D 396 – 48 T. Fuel Oils.

Tentative Methods of Test for:

D 90 – 50 T. Sulfur in Petroleum Products by the Lamp Gravimetric Method,

D 155 – 45 T. Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter,

D 157 – 51 T. Steam Emulsion of Lubricating Oils,

D 613 – 48 T. Ignition Quality of Diesel Fuels by the Cetane Method,

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[8] See p. 361.

[9] See ASTM Viscosity Tables, July, 1953.

D 614 – 49 T. Knock Characteristics of Aviation Fuels by the Aviation Method,

D 909 – 49 T. Knock Characteristics of Aviation Fuels by the Supercharge Method,

D 936 – 51 T. Aromatic Hydrocarbons in Olefin-Free Gasoline by Silica Gel Adsorption,

D 937 – 49 T. Penetration of Petrolatum,

D 972 – 51 T. Evaporation Losses of Lubricating Greases and Oils,

D 976 – 48 T. Color of U.S. Military Gasoline, Automotive (Combat), by Means of an ASTM Color Standard,

D 1015 – 49 T. Measurement of Freezing Points for Evaluation of Purity,

D 1018 – 49 T. Hydrogen in Petroleum Fractions by the Lamp Method,

D 1022 – 49 T. Peroxides in Butadiene (Ferrous-Titanous Method),

D 1091 – 50 T. Phosphorus in Lubricating Oils, Lubricating Oil Additives, and Their Concentrates,

D 1095 – 50 T. Analysis of 60 Octane Number Iso-Octane—Normal Heptane ASTM Knock Test Reference Fuel Blends by Infrared Spectrophotometry,

D 1096 – 50 T. 1,3 Butadiene in C_4 Hydrocarbon Mixtures by Ultraviolet Spectrophotometry, and

D 1157 – 51 T. Total Inhibitor Content (*p*-Tertiary-Butylcatechol) of Butadiene.

AMERICAN STANDARDS

Committee D-2 recommends that the following actions be recommended to ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

(*1*) Consideration of all ASTM Standards which may be revised as a result of recommendations appearing in this report for continuation as American Standard in their revised form.

(*2*) Consideration of Methods D 892, D 1024, D 1088, D 1089, D 1094 and D 1250, for approval as American Standard.

(*3*) Consideration of the proposed replacement for D 721 – 51 T (Z11.52-1952), Oil Content of Petroleum Waxes, (see Appendix IX, 1952 Report of Committee D-2), for approval as American Standard.

(*4*) Consideration of Method D 381 – 52 T, Existent Gum by Jet Evaporation, which replaces D 381 – 50 (Z11.36-1950), for approval as American Standard.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[11]

This report has been submitted to letter ballot of the committee, which consists of 117 voting members; 72 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. L. MAAG,
Chairman.

W. T. GUNN,
Secretary.

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[11] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

- Test for API Gravity of Petroleum and its Products by Hydrometer (D 287 - 53 T), and
- Test for Specific Gravity of Petroleum and its Products by Hydrometer (D 1298 - 53 T).

Revision of Tentative Specifications for:

- Gasoline (D 439 - 52 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentative methods appear in the 1953 Supplement to Book of ASTM Standards, Part 5.

PROPOSED CHANGES IN THE "ASTM MANUAL OF ENGINE TEST METHODS FOR RATING FUELS" (1952 EDITION) FOR THE ELIMINATION OF THE BOUNCING PIN

The bouncing pin is to be eliminated from ASTM Method D 357 - 49, Test for Knock Characteristics of Motor Fuels by the Motor Method, and ASTM Method D 908 - 51, Test for Knock Characteristics of Motor Fuels by the Research Method, and shall not be used after January 1, 1954.

In addition to the revisions in ASTM Methods D 357 and D 908,[1] specific deletions and changes in the ASTM Manual of Engine Test Methods for Rating Fuels are detailed below. In addition, there will have to be a revision in the numbering of all tables and illustrations.

Page vi, Instruments:
- Delete Section 117, "Bouncing Pin and Knockmeter."

Page vii, Motor and Research:
- In Section 310 delete "Bouncing Pin and."
- Delete Section 311, "Adjustments for Non-Suspended Bouncing Pin."
- Delete Section 312, "Adjustments for Shoulder Suspended Bouncing Pin."
- Delete Section 313, "Bouncing Pin Operating Characteristics."

Page x, Instruments:
- In Section 468, change title from "Bouncing Pin" to "Combustion and Injection Indicators."
- Delete Section 470, "Bouncing Pin Thermal Element."

Page xi, Instruments and Exhaust System:
- In Section 618 delete "Bouncing Pin."

Page xii, Methods:
- Delete Fig. 2, "Adjustable Bouncing Pin."

Page xiii:
- Delete Fig. 28, "Shoulder Suspended Bouncing Pin for Motor and Research Units."

Page xv:
- Delete Table I, "Micrometer Settings for Standard Knock Intensity at a Barometric Pressure of 29.92 in. of Mercury—For Bouncing Pin (Motor Method, $\frac{9}{16}$ in. Venturi)."
- Change Table IA to read Table I.
- In Table III delete "—For Bouncing Pin or Detonation Meter."
- In Table IV delete "—For Bouncing Pin or Detonation Meter."
- Delete Table V, "Micrometer Settings for Standard Knock Intensity at a Barometric Pressure of 29.92 in. of Mercury—For Bouncing Pin (Research Method, $\frac{9}{16}$ in. Venturi)."
- Change Table VA to read Table V.

Page xvi, Maintenance:
- In Table XVII delete "Bouncing Pin."

Page 79, Apparatus (Cetane):
- In Section 109(*d*), line 2, delete "bouncing pin or."

Page 86:
- Delete Section 117 in its entirety

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[1] See pp. 357 and 358.

and also the title above it, "Bouncing Pin and Knockmeter."

Page 120:

In Section 221 delete "IA and II" and "VA and VI."

Page 126:

In Line 1 delete "bouncing pin or."

In Section 302(*d*), Line 3, change "bouncing pin" to read "pick-up."

In Section 302(*e*), Line 2, change "bouncing pin" to read "pick-up."

In Section 302(*f*), Line 8, change "bouncing pin" to read "pick-up."

Page 138:

In Fig. 27 delete "F—Knockmeter Fuse—Motor and Research Methods."

Page 140:

In Line 5 delete "Bouncing Pin and" in the Title and omit Sections 310 and 310(*a*).

Change Section 310(*b*) to read 310.

Pages 140 to 144:

Delete Sections 311, 312, and 313 in their entirety and also Fig. 28.

Page 148:

In Section 316(*b*), Line 5, change the sentence to read: "With the $\frac{9}{16}$ in. venturi, Table I is used for the Detonation Meter. Tables III and IV for the $\frac{19}{32}$ and $\frac{3}{4}$ in. venturis, respectively, are used with the Detonation Meter."

Page 148:

In Line 11 delete "Table I—Bouncing Pin—$\frac{9}{16}$ in. Venturi."

Page 148:

In Line 12 delete "Bouncing Pin or."

Page 148:

In Line 14 delete "Bouncing Pin or."

Page 148:

In Line 16 change "Table IA" to read Table I."

Page 148:

In Line 18 change "Four" to read "Three."

Page 148:

In Section 316(*c*), Line 1, delete "for the bouncing pin or table VA."

In Section 316(*e*), Line 1, delete "the contact gap of the bouncing pin or."

In Line 6, delete "contact gap of the bouncing pin or." Delete Section 316(*f*).

Page 149:

In Section 318(*a*), Line 3, delete "and bouncing pin."

In Section 318(*b*), Lines 9 through 11, delete "With the bouncing pin the difficulty may be corrected by adjustment of this part. With both the bouncing pin and Detonation Meter."

Page 150:

In Section 319(*c*), Line 3, delete "IA" and "VA."

Page 238:

In Section 435(*a*), Line 4, delete "bouncing pin."

Page 273, Instruments:

Change title "Bouncing Pin" to read "Combustion and Injection Indicators" and delete the first three lines of Section 468 starting with "Motor and Research."

Page 274:

In Section 468(*b*), Line 5, change "bouncing pin" to read "instrument."

Delete from the title of Table XVII "Bouncing Pin" and "Motor, Research, and."

In Section 468(*d*), Line 2, delete "bouncing."

Page 275:

In Section 469, Line 1, delete "is used with both the bouncing pin and the detonation meter and."

Delete Section 470 in its entirety and its title "Bouncing Pin Thermal Element."

Page 278:

In Table XVIII, and Lines 4, 10

and 13, change "Bouncing pin" to "Instrument hole."

In Line 15 change "Bouncing pin" to "Instrument."

Page 302:

Replace Fig. 85, "End View of Motor Unit" with a similar figure in which the drawing of the Bouncing Pin and its references "B-Knockmeter Thermal Element," "F-Bouncing Pin Terminals," and "K-Bouncing Pin" have been replaced by a reference to, and picture of, the Detonation Pick-up.

Page 308:

Change wiring diagram to eliminate equipment for, and reference to, the "Bouncing Pin," the "Knockmeter Thermal Element," "Fuse," and "Voltage Divider."

Page 322 and 323:

In Section 618 delete "Bouncing Pin" from the title.

Change Paragraph (*a*) to read:

618 (*a*). *Motor, Research, Aviation, and Cetane.*—Remove the wooden plug from the knock instrumentation hole in the cylinder. For Motor and Research units, insert the D-1 pick-up using the gasket supplied (tighten the D-1 pick-up with a torque of approximately 30 lb-ft using a new gasket each time it is installed). Install the detonation meter in accordance with instructions furnished by the supplier. For the Aviation unit, insert the thermal plug, *H*, Fig. 5 (p. 40), and for the Cetane, the combustion indicator, *F*, Fig. 10 (p. 64).

Delete Paragraph (*b*).

Page 333:

Delete from the Index "Bouncing Pin" and all sub-headings under "Bouncing Pin."

Page 334:

In Combustion Indicator, Line 3, delete "Motor, Research."

Page 338:

Under "Knock measuring instrumentation" delete "Bouncing Pin."

Delete "Knockmeter drift, prevention of, Motor, Research, 144."

Under "Micrometer settings, barometric pressure corrections" delete "for bouncing pin, Motor, 10."

APPENDIX I

PROPOSED METHOD OF TEST FOR FUNCTIONAL LIFE OF BALL BEARING GREASES[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

Scope

1. This method of test is intended to evaluate the functional life of ball bearing greases when tested under prescribed laboratory conditions. It is not the equivalent of long-time service tests, and is limited to greases for operating temperatures up to 125 C (257 F).

Apparatus

2. The apparatus required for this test is illustrated in Fig. 1, and is described in detail in the Appendix. New ball bearings of the type specified are required for each test.

Grease Sample

3. Take the sample of grease for test with a minimum of disturbance. Do not reincorporate any free oil that may have separated or include this oil, when taking the sample.

Test Bearings

4. (*a*) The test bearings shall be No. 30BC03 ball bearings of 52100 steel, having pressed-steel retainers. The internal radial clearance shall be 0.0002 to 0.0006 in., and the bearings shall be heat-stabilized for operation up to 160 C.

Preparation of Bearings

5. Clean the bearings immediately before using the test machine; they should not be stored after cleaning. Wash the bearings thoroughly in ASTM Precipitation Naphtha (ASTM Method D 91, Test for Precipitation Number of Lubricating Oils),[3] turning the outer race to remove all traces of slushing compound or oil from races, balls, and separator pockets. Repeat washing, using alcohol Formula 30 or equivalent, and dry by placing in a warm, dust-free oven for several hours. Avoid spinning the bearings when dry. The test housing and end caps should also be washed and dried in preparation for the test.

Procedure for Performance Life, Including Leakage Evaluation

6. This procedure simulates the type of grease-packing commonly encountered in the field, wherein the grease is pumped into the bearing housing with a grease gun.

(*a*) Fill both bearings completely, using a clean spatula to force the grease between walls, separator, and races. Strike off surfaces of the grease level with faces of the inner and outer races.

(*b*) Mount bearings on the shaft with the heater assembly in place, without

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1953.

[3] 1952 Book of ASTM Standards, Part 5.

rotating the bearings. Assemble the lock washers, and turn up lock nuts with the fingers. Slide the shaft assembly carefully into the housing, lining up the slot in the heater assembly with the set screw on the housing. Tighten lock nuts with spanner wrenches as tightly as possible by hand. Bend over one tang of each washer into a slot in the lock nut.

NOTE 1.—In all of these operations, extreme care must be taken that dirt or metal particles or slivers do not get into the grease or housing.

(*c*) Fill each end cap with grease, striking off the surface of the grease between the edge of the inner bore and outer rim.

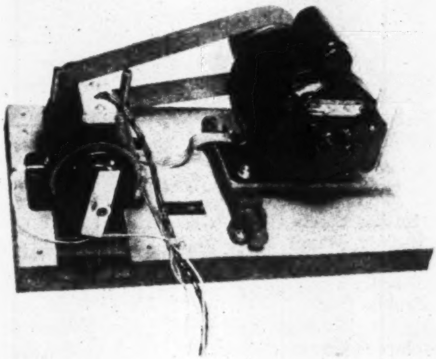

FIG. 1.—Grease Tester with Electric Motor.

(*d*) Place the thrust washer against the face of the bearing opposite the pulley end, and assemble the end caps on that end. Then assemble the end cap on the pulley end, drawing up screws evenly to move the bearing assembly smoothly against action of the thrust spring.

(*e*) Complete the assembly of the apparatus. The control thermostat should be in the well nearest the drive motor, with the limit thermostat in the other well. The spring-loaded thermocouples should be placed in position against the outer bearing rings.

(*f*) Start the driving motor, recording time of start, and adjust the heater transformer to bring the temperature up to control level in approximately 2 hr. Then readjust the heater so that the temperature control relay switches from high heat to low heat about 50 per cent of the time. This setting of the heater transformer shall be maintained throughout the run.

(*g*) At the end of 20 hr running, shut off both the motor and the heater. Measure grease leakage around the shaft hole, and note any leakage from the hole in the bottom of the housing. After 4 hr, again turn on the motor and heater simultaneously. Repeat this cycle of 20 hr on, 4 hr off, until a bearing failure occurs. The cycling may be done manually or by automatic control, as shown in the circuit diagram in Fig. 3 of the Appendix.

Procedure for Performance Life Alone

7. (*a*) In this procedure, one third of the free space in each bearing is filled with grease, while none is placed in the housing. This type of grease packing is typical of many applications, including factory-packed bearings.

(*b*) Pack each of the two bearings with 6.0 ± 0.1 g of grease. Work the grease uniformly into both sides of each of the bearings with a narrow blade spatula, frequently turning the outer bearing ring with respect to the inner ring.

(*c*) Complete the test as prescribed in Section 6(*b*) to (*g*) for assembly of the unit and method of operation.

Results

8. (*a*) In either of the two procedures (Sections 6 and 7), the earliest positive evidence of bearing distress shall determine the time of failure. This may occur in one of the following ways:

(*1*) Stalling of the motor during operation,

(*2*) Stalling of the motor when attempting to start after shutdown, (*3*) By a temperature rise of 10 C above the control temperature, or

(*4*) By a distinct increase in the noise level which persists for 10 min, either intermittently or continuously.

Failure by the means indicated in items (*3*) or (*4*), shall be based on a repetition of the occurrence a second time from a cold start. Smoking of the heater shall not be considered a failure, nor cause to shut down the unit.

(*b*) After termination of the test, disassemble the unit, making sure that the spring-loaded thermocouples and heater-assembly set screw are removed before attempting to slide out the shaft assembly. Then make a careful inspection of the unit, noting the grease leakage, the amount of grease left in the housing (in the case of the leakage-evaluation procedure given in Section 6), and the condition of the test bearing.

Report

9. Report the results of the inspection, together with the test conditions, on the report form shown in Table I.

Precision

10. The nature of this method is such that wide variations in running time between individual tests are typical. A statistical analysis indicates that a minimum of three tests should be run. Reproducibility of the method approximates ±35 per cent of the mean functional life.

TABLE I.—SUGGESTED REPORT FORM FOR FUNCTIONAL BALL BEARING GREASE TEST.

Test Conditions:

Grease: ____________________
Date: Start __________ Finish __________
Total Running Time: ____________________
Amount of Grease and Packing Method: ______
____
Temperature of Test Bearing: __________ F.
Cycle: _____ Hours on _____ Hours off ______
Total Cycles per Week: ____________________
Radial Loading: Motor ______ Other ______
Heater Transformer Setting: __________ Volts
Noise (Type and Time of Occurrence): ______
____
____

Type of Failure:

None .... ________
Stalled While Running .... ________
Stalled on Starting .... ________
Excessive Temperature Rise ..... ________
Excessive Noise .... ________
Other: ____________________

Inspection of Test Bearing After Test:

Rotates Freely .... ________
Slightly Rough .... ________
Rough .... ________
Seized .... ________
Broken Cage .... ________

Leakage of Grease:

	First Cycle	Total
Leakage around Shaft Hole (Pulley End)	______	______
Leakage under Housing....	______	______
Amount of Grease Left in End Bell (Pulley End)... (Leakage Evaluation Procedure)	______	______

APPENDIX

APPARATUS[4]

Grease Tester:

A1. (*a*) The grease tester shall be a small, belt-driven, motor-like unit with two bearings, one for test, and one for a guide. The construction is shown in Fig. 2. The main housing shall be precision-bored to insure correct bearing alignment. The two end caps serve as grease retainers for the bearing, simulating the arrangement in an electric motor.

(*b*) A hole, $\frac{1}{2}$ to $\frac{3}{4}$ in. in diameter, shall be provided at the bottom of the housing to permit excess grease and oil to drain without interfering with the electric heater. A pan, 3 in. wide by 9 in. long by ½ in. deep, to collect this drainage, shall be placed in the groove of the base supporting the tester.

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[4] Detailed drawings of this apparatus are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

(*c*) The shaft has two shoulders to insure proper positioning of the bearings. The test end of the shaft shall be keyed for the driving pulley, while the guide end is reduced in diameter and threaded for a small cooling fan. The cooling fan keeps the guide bearing about 5 C cooler than the test bearing.

NOTE 2.—The guide bearing failed in a number of the tests conducted, and consideration is being given to its inclusion as an additional test bearing.

Drive:

A3. The unit shall be driven at approximately 3500 rpm by a ¼-hp, 1725-rpm electric motor. A suitable pulley and belt are required. A flat- or Vee-belt drive is satisfactory, the latter being more quiet. The motor shall be mounted on a hinged base, and adjusted so that its weight applies a radial load of 25 ± 5 lb on the test bearing.

Control:

A4. (*a*) A method of temperature control shall be obtained by incorporating a 20-ohm resistor in series with the electric heater in the test unit, as shown in the circuit diagram in Fig. 3. This resistor is switched in and out of the circuit by the 125 C control thermostat (sealed-in-glass mercury type), located on the loaded side of the test bearing. This provides periods of low and high heat to keep the unit operating at 125 C. When lubrication becomes so inadequate that the frictional heat plus the low electrical heat raises the bearing temperature to cause contact in the 135 C thermostat, the power to the unit is shut off and the test stops. In this way the 135 C thermostat, located on the unloaded side of the bearing, acts as a limit switch to prevent damage to the unit when bearings are in distress. Zinc fuses are also

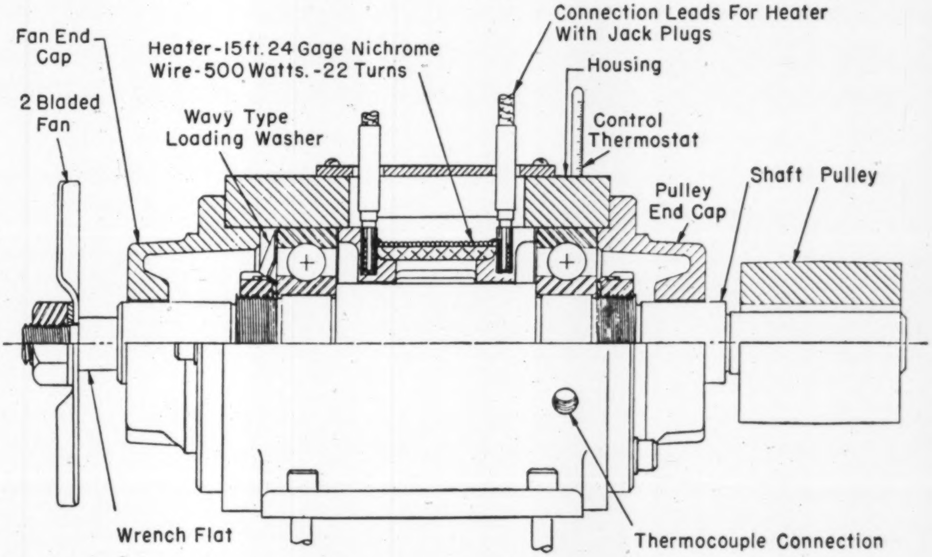

FIG. 2.—Assembly of Grease Tester.

(*d*) Spanner nuts, with keyed multilug lock washers, keep the inner race of the bearing tight on the shaft. A thrust load of 40 ± 5 lb may be obtained by use of a Z-spring. This is necessary to make the balls rotate in the desired manner.

Electric Heater:

A2. An electrical heater shall be wound on an insulated open spool surrounding the center of the shaft. The heat is conducted both to the housing and to the shaft in much the same manner as heat flows in an electric motor. The heater shall have a rating of 500 w at 110 v, and be made of 15 ft of No. 24 Nichrome V wire, or its equivalent. A set-screw in the housing fits into a longitudinal slot in the heater frame to keep it from turning.

provided for protection in case of a stall, overload, or short-circuit.

(*b*) At the start of the test, line switch S_1 and motor switch S_2, Fig. 3, are closed. The start button is used to energize contactor K_1 and close contact K_1. The variable transformer is adjusted to that voltage which causes the relay K_4 to be on about 50 per cent of the time after the unit has been operating 2 hr. Two pilot lights are shown in the circuit that indicate the operation of K_4 relay, and are obviously of great help in setting the transformer. When 125 C is reached, the mercury thermostat closes, energizing relay K_3, opening normally closed contact K_3, dropping out contactor K_4, and opening contact K_4. This puts the 20-ohm resistor in series with the 500 w heater in the test unit thus lowering the heat input.

Time Meter:

A5. A time meter in parallel with the drive motor shall be used to register the hours of operation.

NOTE 3.—An electronic relay used as indicated in the circuit diagram (Fig. 3) serves to reduce the current carried by the mercury thermostats and, in this way, minimizes oxidation of mercury within the thermostat, prolonging thermostat life.

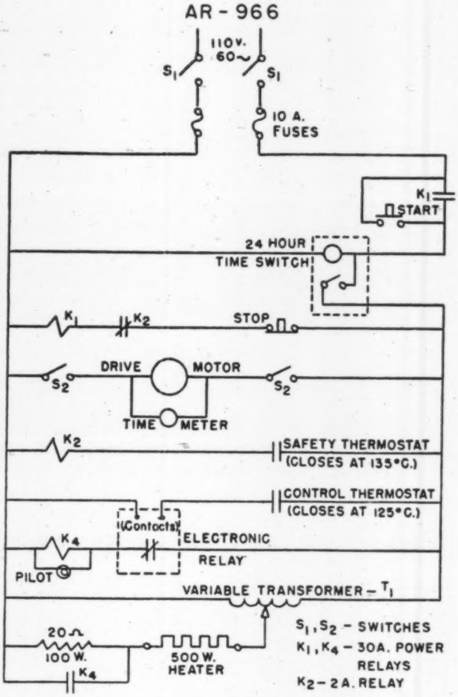
AR - 966
110 V.
60~
S1
S1
10 A.
FUSES
K1
START
24 HOUR
TIME SWITCH
K1
K2
STOP
S2
DRIVE
MOTOR
S2
TIME
METER
K2
SAFETY THERMOSTAT
(CLOSES AT 135°C.)
CONTROL THERMOSTAT
(CLOSES AT 125°C.)
(Contacts)
K4
ELECTRONIC
RELAY
PILOT
VARIABLE TRANSFORMER – T1
20 Ω
100 W.
500 W.
HEATER
K4
S1, S2 – SWITCHES
K1, K4 – 30A. POWER
RELAYS
K2 – 2 A. RELAY

Fig. 3—Circuit Diagram of Grease Tester.

APPENDIX II

PROPOSED METHOD OF TEST FOR CHLORINE IN LUBRICATING OIL[1, 2]

SODIUM ALCOHOLATE VOLUMETRIC METHOD

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method[3] describes a procedure for determination of the chlorine content of lubricating oils and greases with which chlorinated additives have been blended. It is equally applicable to new or used oils and greases and to the additive concentrate. The procedure assumes that bromine and iodine will not be present.

NOTE 1.—A method based on bomb combustion for analysis of the same type of material is described in ASTM Method D 808,[4] Test for Chlorine in Lubricating Oils and Greases by the Bomb Method.

Outline of Method

2. The sample, dissolved in a low boiling hydrocarbon mixture, is boiled under reflux with metallic sodium and *n*-butyl alcohol. Under these conditions the chlorine is converted to sodium chloride which is then extracted with water. The chloride in the extract is determined volumetrically by titration with silver in the presence of thiocyanate.

Apparatus

3. *Reaction Vessel* consisting of a 300-ml round-bottom flask with standard-taper joint and surmounted with a water-cooled condenser.

Reagents

4. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

(*a*) *Ferric Alum Indicator Solution.*—Dissolve 350 g of $Fe_2(SO_4)_3 \cdot (NH_4)_2 \cdot SO_4 \cdot 24H_2O$ in 1 liter of distilled water and add 200 ml of aerated HNO_3 (30 per cent).

(*b*) *n-Butyl Alcohol.*

(*c*) *Naphtha,* paraffinic, distilling between 210 and 250 F.

(*d*) *Nitric Acid* (*approximately 30 per cent by weight*).—To prepare approximately 1 liter of solution, mix 345 ml of HNO_3 (sp gr 1.42) with 690 ml of distilled water. Aerate to remove oxides of nitrogen.

(*e*) *Nitrobenzene* (*technical*).—The nitrobenzene used must not show a visible precipitate when tested with alcoholic $AgNO_3$ solution.

(*f*) *Phenolphthalein Indicator Solution,* 1 per cent in 60 per cent isopropyl alcohol.

(*g*) *Standard Silver Nitrate Solution* (*0.1 N*).—Dissolve 17.0 g of $AgNO_3$ in

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1953.

[3] This method is technically equivalent to the Institute of Petroleum Method No. 118/48.

[4] 1952 Book of ASTM Standards, Part 5.

distilled water and dilute to 1 liter. To standardize this solution, weigh 0.20 to 0.25 g (weighed to 0.1 mg) of NaCl (previously dried to constant weight at 120 C). Transfer to a 500-ml Erlenmeyer flask, and dissolve in 100 ml of distilled water. Titrate with the $AgNO_3$ solution, following the procedure described in Section 5(*f*) and (*g*). Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality of AgNO}_3\text{ solution} = \frac{\text{mg of NaCl}}{\text{ml of AgNO}_3 \times 58.45}$$

(*h*) *Sodium Metal.*

(*i*) *Standard Thiocyanate Solution (0.05 N).*—Dissolve 3.80 g of NH_4CNS or 4.85 g of KCNS crystals in distilled water and dilute to 1 l. To standardize this solution, measure 40 ml into a flask containing 5 ml of HNO_3 (30 per cent) and 50 to 60 ml of water. Swirling the flask constantly, add 22 to 24 ml of 0.1 *N* $AgNO_3$ solution from a buret; then stopper and shake for 20 to 30 sec. Add 2 to 3 ml of ferric alum indicator solution and back-titrate with the thiocyanate solution to the red $Fe(CNS)_3$ end point. Calculate the normality of the thiocyanate solution as follows:

$$\text{Normality of thiocyanate solution} = \frac{\text{ml of AgNO}_3 \times N\text{ of AgNO}_3}{\text{ml of thiocyanate}}$$

Procedure

5. (*a*) Shake the sample, if liquid, to ensure homogeneity. Weigh a portion, estimated to contain 0.05 to 0.1 g of chlorine, into the 300 ml flask. Add 25 ml of naphtha and swirl until the sample is dissolved.

Reaction and Extraction

(*b*) To the solution of the sample, add 5 ml of *n*-butyl alcohol and 1.5 ± 0.1 g of freshly cut sodium. Connect the flask to the condenser and boil the liquid vigorously under reflux for 1 hr. Introduce an additional 20 ml of *n*-butyl alcohol through the top of the condenser and continue refluxing until no metallic sodium remains.

(*c*) Add 25 ml of distilled water to the flask through the condenser, cool to room temperature, and transfer the mixture to a 500-ml separatory funnel. Wash the flask with 50 ml of HNO_3 (30 per cent) and then with 50 ml of the naptha, adding these washings to the separatory funnel. Adjust the acidity of the aqueous layer until just acid to phenolphthalein, and then add 5 ml more HNO_3 (30 per cent).

(*d*) Shake the funnel and contents, and, after settling, draw the aqueous layer into a clean 500-ml glass-stoppered Erlenmeyer flask. Wash the original 300-ml reaction flask with 25 ml of distilled water, transfer to the separatory funnel, and wash the naphtha layer; transfer this washing to the 500-ml flask. Repeat with a second 25-ml portion of water.

Chloride Determination

(*e*) Boil the combined acidic aqueous extracts and washings until free of H_2S, as detected by dipping a piece of lead acetate test paper into the solution (no black discoloration). Filter through a qualitative filter paper and collect in a second 500-ml glass-stoppered Erlenmeyer flask. Cool the solution to room temperature.

(*f*) After the solution has reached room temperature, add 10 ml of nitrobenzene (**Caution,** Note 2; Note 3), 3 ml of ferric alum indicator and, from a buret, 0.4 to 0.6 ml of 0.05 *N* thiocyanate solution. Swirling constantly, titrate with 0.1 *N* $AgNO_3$ solution until the red color of the $Fe(CNS)_3$ is discharged; then add 2 to 5 ml in excess.

NOTE 2: **Caution.**—Care should be exercised to avoid breathing fumes of nitrobenzene, which are toxic.

NOTE 3.—Nitrobenzene not only is efficient as a coagulant for the AgCl but removes it from the aqueous phase. Alternatively, isoamyl alcohol may be used to coagulate the precipitate, or the AgCl may be removed by filtration after adding a slight excess of $AgNO_2$ and washing with dilute acid.

(*g*) Stopper the flask tightly and shake vigorously for 15 sec to coagulate and remove the precipitate from the aqueous phase (**Caution,** Note 4). Without refilling the buret, titrate slowly with 0.05 *N* thiocyanate solution until the end point is approached, as indicated by a more slowly fading red color. Stopper the flask, shake vigorously for 20 to 30 sec, and continue the titration until one drop produces a distinct reddish coloration that does not fade upon swirling or vigorous shaking.

NOTE 4: **Caution.**—Care should be exercised in opening the flask after shaking. Pressure may develop, causing small amounts of the acid solution to be sprayed from the mouth of the flask as the stopper is removed.

Blank Determinations

(*h*) Simultaneously with each set of sample determinations, make duplicate blank determinations, following exactly the procedure used for the sample determinations (except for omission of the sample). Always use identical reagents for the blank determination and for the sample determination. If the titration exceeds 0.05 ml, discard the responsible reagent.

Calculation

6. Calculate the total chlorine content of the sample as follows:

$$\text{Chlorine, per cent by weight} = \frac{(VN - vn) \times 35.46}{10W}$$

where:

V = milliliters of $AgNO_3$ solution used,
N = normality of the $AgNO^3$ solution,
v = milliliters of thiocyanate solution used,
n = normality of the thiocyanate solution, and
W = grams of original sample used.

Precision

7. The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatibility.*—Duplicate results by the same operator should not differ by more than the following amounts:

Chlorine Content, per cent	Repeatability
0.1 to 1	0.03
over 1	3 per cent of amount present

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Chlorine Content, per cent	Reproducibility
0.1 to 1	0.04
over 1	6 per cent of amount present

APPENDIX III

PROPOSED METHOD OF TEST FOR SODIUM IN RESIDUAL FUEL OIL BY FLAME PHOTOMETER[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of sodium in residual fuel oil by means of a flame photometer.

Outline of Method

2. A weighed sample is reduced to a carbonaceous ash under controlled conditions. The residual carbon is removed by heating in a muffle furnace at 550 C. The ash is dissolved and diluted to volume according to the respective flame photometric methods for sodium employing either the Beckman flame photometer or the Perkin-Elmer flame photometer.

Purity of Reagents and Water

3. Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. References to water shall be understood to mean distilled water.

METHOD A. BECKMAN FLAME PHOTOMETER

Apparatus

4. (*a*) *Beckman Flame Photometer,* Model 10300.

(*b*) *Platinum Dish,* 100-ml capacity, approximately 3.5 cm in depth.

(*c*) *Electric Muffle Furnace,* capable of operating over a variable range of 200 to 600 C and of maintaining a temperature of 550 ± 50 C.

Reagents

5. (*a*) *Standard Sodium Solution* (*1000 mg Na per l*).—Dissolve 2.5418 g of dried NaCl in water and dilute to 1 liter.

(*b*) *Hydrochloric Acid* (*sp gr 1.19*).

(*c*) *Hydrofluoric Acid* (*48 per cent*).

Preparation and Standardization of Apparatus

6. (*a*) Dilute the 1000 mg per l standard sodium solution so as to obtain solutions containing, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 15, 18, and 20 mg per l.

NOTE 1.—The use of the standard addition technique, to be described, requires that a linear relationship exist between concentration and intensity of emission. It has been shown that for sodium, a linear relationship exists only in the low concentration range. The solutions described in Paragraph (*a*) are used to determine this characteristic for each instrument. In cases where a linear relationship does not exist for the particular instrument (for example, where hydrogen or acetylene is burned), it is advisable to use a calibration curve, and to check it frequently.

(*b*) Prepare the flame photometer for use as described in the manufacturer's

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1953.

instruction manual. Using the blue sensitive phototube, light the burner and adjust the gas and oxygen so that a blue flame is obtained. Set the selector switch at the 0.1 position and the wavelength dial to correspond to the center of the sodium peak at or near 589.6 mμ, judging by the response of the instrument. Adjust the sensitivity knob of the spectrophotometer so that it will be in the middle of its range (five turns from either the clockwise or counterclockwise positions). Vaporize the 4 mg per 1 standard sodium solution, at the same time adjusting the slit width so that the scale reading (upper or transmission scale) will be approximately 38. Vary the gas rate until a maximum scale reading is obtained. Then without changing the gas rate, adjust the oxygen pressure to obtain the highest scale reading possible. Finally readjust the sensitivity setting until the 4 mg per 1 solution gives a scale reading of 38.5. Typical settings employed with one instrument are as follows:

Propane	6.25 cu ft per hr
Oxygen	40 in. of water
Slit width	0.15 mm
Sensitivity setting	4.5 turns from the counter-clockwise limit
Vaporizing air	20 psi

(*c*) Vaporize each of the standard solutions, checking the 4 mg per 1 standard before each reading to determine whether the instrument is functioning properly. A slight adjustment of the dark current and sensitivity controls is all that should be required.

(*d*) Prepare a working curve by plotting the milligrams of sodium per liter against the scale reading. Use this curve to determine the upper concentration limit of the linear relationship between concentration and scale reading.

Sample Preparation

7. (*a*) In sampling the bulk material or plant stream for which the ash determination is desired, use a clear glass bottle and collect a sample of the approximate size specified in Table I. If duplicate or triplicate determinations are desired, bottle a separate sample for each determination.

NOTE 2.—It is often very difficult to render the contents of a bottle of sample homogeneous. Also, in some cases apparent ash content is reduced as a result of particles adhering tightly to the walls of the container. The adsorption of inorganic ions by glass often is a serious factor when metals are to be determined in the ash. For these reasons, the whole sample must be used for the determination, and the bottle rinsed as described in Paragraph (*f*) if accurate results are to be obtained.

TABLE I.—SAMPLE SIZE.

Ash Content, per cent by weight	Sample Size, g	Required Weighing Accuracy, g
Under 0.001	500 to 1500	1
0.001 to 0.01	300	1
0.01 to 0.1	100	0.5
Over 0.1	25	0.1

Results of somewhat doubtful accuracy may be obtained for samples not bottled in accordance with Paragraph (*a*) by using the following procedure: By shaking or stirring the sample, make it as homogeneous as possible. If necessary, heat to reduce the viscosity. Pour the approximate amount indicated in Table I into a clean, clear glass bottle of appropriate size. Proceed in accordance with Paragraph (*c*).

(*b*) Heat a clean 100-ml platinum dish (Note 3) at 600 C for 10 min or more. Cool to room temperature in a desiccator containing anhydrous calcium sulfate (Drierite), and weigh to 0.1 mg. Repeat the ignition and weighing until successive results do not differ by more than 0.2 mg.

NOTE 3.—The platinum dish must be handled only with platinum tipped tongs. It must never be touched with the fingers during the determination. Before weighings are made, the bottom of the dish should be carefully dusted with a clean camel-hair brush.

(*c*) Carefully clean and dry the outside surface of the sample bottle, and weigh the bottle and contents to within the accuracy specified in the third column of Table I. Transfer approximately 65 ml of sample to the weighed platinum dish. Support the dish on a silica triangle, and heat with a Bunsen burner until the contents ignite and burn readily (Note 4). Continue heating with the burner in such a manner that the sample burns at a uniform and moderate rate and only ash and carbon remain after burning ceases (Note 5). When the flame has gone out, fill the dish approximately two-thirds full with additional sample, and repeat the burning procedure as above. Continue adding and burning the sample until the entire contents of the bottle have been consumed. In transferring the last of the sample, rinse out the last traces from the bottle with three 20-ml portions of xylene, and add the xylene washings to the dish. *CAUTION.—Since xylene is highly flamable, allow the platinum dish to cool before adding the washings.* Dislodge any particles adhering to the walls of the bottle either by vigorous shaking with the xylene, or if necessary by using a plastic-tipped policing rod.

NOTE 4.—If the sample contains an appreciable amount of water, as indicated by spattering when heated, it is advisable to add a few milliliters of absolute alcohol before heating.

NOTE 5.—Special precautions should be taken to guard against undue exposure and possible contaminations from the hood in which the burning is done.

(*d*) Place the dish in a muffle furnace at 200 C (Note 6). Slowly raise the temperature to 550 ± 50 C. Leave the muffle door slightly ajar until little carbon remains in the dish; then close the door and continue the ignition until no carbon is visible. Cool the dish to room temperature in a desiccator containing anhydrous calcium sulfate (Drierite), and weigh to 0.1 mg (Note 3). Continue the ignition for additional 20-min intervals until successive weighings agree to within 0.3 mg.

NOTE 6.—The platinum dishes should be placed on silica plates or silica triangles on the floor of the muffle furnace. Particular care must be exercised to avoid contamination of the sample with particles from the roof, walls, and door of the furnace. During the initial ignition, the opening of the muffle door must be carefully adjusted so that the air flow into the muffle is not excessive. Too great an air flow causes high temperatures in the burning carbon and also possible loss of ash from the dish.

(*e*) Add about 1 ml of HF to the platinum dish and evaporate to dryness to remove silica (Note 7).

NOTE 7.—Unless the silica is removed, low values will result from the occlusion of sodium in the insoluble residue.

(*f*) Rinse the sample bottle with three 10-ml portions of HCl (1:9) and transfer these successive rinses quantitatively to the ashed sample in the platinum dish. Carefully drain and dry the empty sample bottle. Reweigh it with the accuracy prescribed by Table I. Subtract the weight of the empty sample bottle from that of the full sample bottle to obtain the sample weight.

(*g*) Heat the platinum dish on a steam bath, to effect solution of the sodium salts, until 1 ml of liquid remains. Remove from the steam bath, add 5 to 10 ml of water, and filter, using an acid-washed filter paper, into a 100-ml volumetric flask. Wash the dish and filter paper with water, collecting the wash water in the volumetric flask. Dilute to volume with water and mix. Should the ash sample contain less than 5 to 10 mg of sodium, vary the dilution and acid concentration accordingly.

Procedure

8. (*a*) Put the flame photometer into operation, using the same air, gas, oxygen, and instrument settings that were determined during standardization in accordance with Section 6 (*b*). Vaporize

the 4 mg per 1 standard solution and adjust the sensitivity control to give the same scale reading that was obtained when the curve was prepared.

(*b*) Dilute an aliquot of the sample solution so as to obtain a working solution that will give a scale reading near the mid-point of the linear portion of the curve prepared in accordance with Section 6 (*b*). An estimate of the dilution required may be made from the ash value obtained in accordance with Section 7 (*d*) (also, see Note 8).

NOTE 8.—An estimate of the extent of dilution necessary may be obtained by vaporizing a portion of the sample solution and measuring the intensity of the emission with the selector switch in the 1.0 position. By referring to the curve prepared as described in Section 6(*b*) and multiplying the concentration by ten, concentrations up to approximately 100 mg per l may be estimated. The concentration of sodium in the sample solution may also be estimated by referring to a curve prepared by plotting scale reading against sodium concentration of solutions containing 0 to 100 mg per l. In this case, the selector switch should be in the 0.1 position and the slit width set at 0.03 mm.

(*c*) Pipet two 10-ml portions of the working solution into dry flasks. To one, add 10 ml of water and mix. To the other, add 10 ml of a standard solution containing 4 mg Na per l and mix. Vaporize each solution and record the scale reading. Vaporize the 4 mg per l standard sodium solution between each sample solution to check the performance of the instrument.

(*d*) Determine and record the scale reading produced when distilled water is vaporized into the flame.

(*e*) Perform a blank determination on all of the reagents, including the distilled water, in accordance with the procedure just described. Calculate the sodium concentration as described in Section 9.

Calculation and Report

9. (*a*) Calculate the sodium concentration, in parts per million, as follows:

$$\text{Sodium, mg} = \frac{A - B}{C - A} \times \frac{DE}{1000} \times \frac{G}{F} - J$$

$$\text{Sodium, per cent} = \text{mg of sodium} \times \frac{100}{1000W}$$

$$\text{Sodium, ppm} = \text{per cent sodium} \times 10{,}000$$

(*b*) If a calibration curve is used, calculate the sodium concentration, in parts per million, as follows:

$$\text{Sodium, mg} = \frac{H}{I - H} \times \frac{DE}{1000} \times \frac{G}{F} - J$$

$$\text{Sodium, per cent} = \text{mg of sodium} \times \frac{100}{1000W}$$

$$\text{Sodium, ppm} = \text{per cent sodium} \times 10{,}000$$

where:

A = scale reading of 10 ml of working solution plus 10 ml of water,
B = scale reading of distilled water,
C = scale reading of 10 ml of working solution plus 10 ml of standard sodium solution,
D = concentration of standard sodium solution, in milligrams per liter,
E = volume, in milliliters, to which ashed sample is originally diluted,
F = aliquot of original sample solution removed for dilution, in milliliters,
G = volume, in milliliters to which aliquot F is diluted to prepare the working solution,
H = milligrams per liter of sodium in 10 ml of working solution plus 10 ml of water, as determined from the calibration curve,
I = milligrams per liter of sodium, determined from the calibration curve, in 10 ml of working solution plus 10 ml of standard sodium solution,
J = milligrams per liter of sodium in reagent blank, as determined from calibration curve, and
W = grams of sample used.

(*c*) Report results to the nearest 1 ppm.

Precision

10. See the Supplement to this method.

METHOD B. PERKIN-ELMER FLAME PHOTOMETER

Apparatus

11. (*a*) *Perkin-Elmer Photometer*, internal standard type equipment.

NOTE 9.—Details of this method as written are based on the Perkin-Elmer flame photometer, Model No. 52A.

(*b*) *Platinum Dish,* 100-ml. capacity, approximately 3.5 cm in depth.

(*c*) *Electric Muffle Furnace,* capable of operating over a variable range of 200 to 600 C and of maintaining a temperature of 500 ± 50 C.

Reagents

12. (*a*) *Internal Standard Lithium Chloride Stock Solution* (*1000 ppm Li*).—Dissolve 6.1 g of LiCl per liter of water. This solution is equivalent to approximately 1000 mg of lithium per liter (see Note 10).

(*b*) *Standard Sodium Solution* (*400 ppm Na*).—Dissolve 1.0165 g of dried NaCl per liter of water; this solution should contain 10 mg of sodium per 25 ml.

(*c*) *Hydrochloric Acid* (*sp gr 1.19*).

(*d*) *Hydrofluoric Acid* (*48 per cent*).

Standardization of Apparatus and Preparation of Calibration Curves

13. (*a*) Dilute the 400 ppm standard sodium solution to obtain solutions containing 1, 2, 3, 4, 5, 6, 8, and 10 mg of sodium, together with 100 mg of lithium, per liter.

NOTE 10.—The solutions to be analyzed must contain no more of the element to be determined than the highest standard and must contain the same concentration of lithium as the standardizing solutions. It is important that both standards and unknowns be made up to the desired lithium concentration from the same lithium stock solution.

(*b*) Prepare the flame photometer according to the manufacturer's instruction manual. Turn on the acetylene gas to 3 psi and light the burner. Set the air pressure to 10 psi and increase the acetylene pressure to 5 psi. Set the internal standard dial to 0 and the coarse gain control to maximum. Turn the zero adjustment knob until the meter reads 50 (center scale position). Tune the wavelength selector dial to the element to be determined. Pour into the atomizer the most concentrated standard solution to be used and turn the wavelength selector dial above and below the position marked for the element to be determined. As the correct wavelength is passed, the meter needle swings to the right. If the meter needle goes off scale, turn the coarse gain control counterclockwise until the needle remains on the scale at the wavelength giving maximum deflection.

(*c*) With the internal standard dial set at 100, adjust the coarse and fine gain controls, using the 10 mg per l standard sodium solution, until the meter returns to 50. With no sample in the atomizer and the internal standard dial set to 0, reset the meter adjustment knob to 50. Repeat the foregoing operations and make adjustments if necessary. Pour in the second standard solution (8 mg per l), bring the meter back to 50 by turning the internal standard dial, and record its balance position.

(*d*) Atomize the first standard solution and bring the meter back to 50 with the internal standard dial, record its balance position. If the reading differs by more than one division, reject the value obtained and repeat the foregoing operations until consistent readings are obtained.

(*e*) Repeat the procedure in Paragraph (*d*) for each of the standard solutions and plot a calibration curve of internal standard dial readings *versus* concentration of sodium in milligrams per liter.

Sample Preparation

14. (a) Proceed in accordance with Section 7 (*a*) to (*f*).

(*b*) Heat the platinum dish on a steam bath, to effect solution of the sodium salts, until 1 ml of liquid remains. Remove from the steam bath, add 5 to 10 ml of water, and filter, using an acid-washed filter paper, into a 100-ml volumetric flask. Wash the dish and filter paper with water, collecting the wash water in the volumetric flask. Add 10 ml of the LiCl stock solution, dilute to volume with water, and mix. Should the ashed sample contain less than 5 to 10 mg of sodium, vary the dilution and the acid and lithium concentrations accordingly.

Procedure

15. (*a*) Adjust the flame photometer for operation according to the directions given under the standardization of apparatus (Section 13(*b*)). Check the calibration curve with the 10 mg per l and the 3 mg per l standard sodium solutions. Atomize the unknown sample solution and record the internal standard dial reading to obtain the meter balance as 50. From the calibration curve, determine the concentration of sodium in milligrams per liter.

(*b*) Perform a blank determination on all of the reagents, including the distilled water, in accordance with the preceding paragraph.

(*c*) If the concentration of the unknown samples exceeds 10 mg of sodium per liter an aliquot of the diluted sample may be selected on the basis of the percentage ash determination in Section 14 to find a working solution in the proper range. This selected aliquot, adjusted for lithium content, diluted to volume, and mixed, may be atomized to find a suitable internal standard dial reading.

(*d*) *Interferences Using the Internal Standard Procedure:*

(*1*) *Potassium Interference in Sodium Determination.*—The effect of the presence of potassium on sodium measurements has been found to be negligible with concentrations up to 10 mg per l of potassium and with sodium concentrations of 0 to 10 mg per l.

(*2*) *Iron Interference on Sodium.*—There is no interference effect from iron at a concentration up to 25 ppm when sodium is present at 5 ppm (see Table II).

TABLE II.—IRON INTERFERENCE WITH SODIUM PRESENT AT 5 PPM CONCENTRATION.

Fe^{+++}, ppm	Approximate Error for Na Measurement, per cent
1000	66
500	29
250	16
100	..
50	5
25	0

Calculation and Report

16. (*a*) Calculate the sodium concentration, in parts per million, as follows:

$$\text{Sodium, mg} = \frac{(A - B) \times C \times E}{1000D}$$

$$\text{Sodium, per cent} = \text{mg of sodium} \times \frac{100}{1000W}$$

$$\text{Sodium, ppm} = \text{per cent sodium} \times 10{,}000$$

where:

A = scale reading for the sample, expressed as miligrams per liter,
B = scale reading for reagent blank, expressed as milligrams per liter,
C = volume, in milliliters, to which ashed sample is originally diluted,
D = volume, in milliliters, of aliquot taken from original dilution volume,
E = volume, in milliliters, to which aliquot D is diluted,
W = grams of sample used.

(*b*) Report results to the nearest 1 ppm.

Precision

17. See the Supplement to this method.

SUPPLEMENT

SUMMARY OF RESULTS OF COOPERATIVE TESTS

The results of cooperative tests on this method for sodium, and a statistical analysis of the data, are given in Table III.

TABLE III.—SUMMARY OF RESULTS OF COOPERATIVE TESTS ON THE FLAME PHOTOMETRIC METHOD FOR SODIUM.

Laboratory	Sodium, ppm					
	Sample E-1	Sample E-2	Sample E-3	Sample E-4	Sample E-5	Sample E-6
A	16	44	0		30	81
	19	46	0		27	80
	18	43			28	79
Avg	18	44	0		28	80
B				19		
	25	46	0	20	37	94
	25	44	0	20	38	95
	23	42	1	21	37	97
Avg	24	44	0	20	37	95
C	26	47	0		33	79
	25	49	1		35	78
	25	48	0		36	80
Avg	25	48	0		35	79
D	23	45	0	19	28	76
	24	46	0	15	26	80
	23	42			29	81
Avg	23	44	0	17	28	79
E	22	38	0	15	25	88
	23	37	0	15	27	91
Avg	23	38	0	15	26	90
F				17		
	22	44	0	20	41	112[a]
	23	41	0	15	38	118[a]
	23	38	0	17	39	118[a]
Avg	23	41	0	17	39	116[a]
G				22		
				20		
	24	43	1	20	27	80
	25	47	0	20	28	79
	25	47	0	20	27	79
Avg	25	46	0	20	27	79
H	25	47	0	20		
	30	46	0	17		
Avg	28	46	0	18		
K				22		
	25	50	0	23	23	80
	26	51	0	22	25	77
	24		1	23	23	77
Avg	25	51	0	23	24	78
Summary[b]						
N	25	24	23	23	23	20
P	24	45	0	19	31	83
%	3	4	..	3	6	6
$100\%/\bar{P}$	13	9	..	16	19	7

[a] Value not included in statistical analysis.

[b] The symbols used in this summary are defined as follows:

N = Number of individual determinations averaged,

P = Arithmetic average,

% = Standard error of an individual determination (root mean square), and

$100\%/\bar{P}$ = Percentage variation.

APPENDIX IV

PROPOSED RECOMMENDED PRACTICES FOR APPLYING PRECISION DATA GIVEN IN ASTM METHODS OF TEST FOR PETROLEUM PRODUCTS AND LUBRICANTS[1, 2]

This is a proposed recommended practice and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This recommended practice describes ways in which the precision limits can be used for rejection of faulty test results by an operator and the limits within which the average should be. It is also intended to indicate when results obtained by two laboratories differ by an amount sufficient to be considered suspect, and the limits within which the correct value should be expected.

(*b*) This practice applies only to those ASTM methods specifically referring to it and is intended to apply at a confidence level of 95 per cent. These recommendations assume that the method is under control by the user and that the material tested behaves in a manner similar to that on which the precision data of the applicable method was obtained.

NOTE 1.—For recommended practices on Designating Significant Places in Specified Limiting Values, see ASTM Standard E 29.[3]

Definitions

2. Variability[3, 4] is a measure of the inherent random errors of the test operation. The most efficient measure of variability for a normal distribution is the standard deviation. In ASTM methods of test for petroleum products and lubricants, it has been judged useful to evaluate the variability for two different situations as follows:

(*a*) *Repeatability* is a quantitative measure of the variability associated with a single operator in a given laboratory, generally with the same apparatus and within a small interval of time. It is defined as the greatest difference between two single and independent results that can be considered acceptable (not significantly different) at the 95 per cent probability level (for methods referring to this recommended practice).

(*b*) *Reproducibility* is a quantitative measure of the variability associated with operators working in two different laboratories. It is defined as the greatest difference between a single test result obtained in one laboratory and a single test result obtained in another laboratory that need not be considered suspect (significantly different) at the 95 per cent probability level (for methods referring to this recommended practice).

NOTE 2.—The definition for Reproducibility has been based on the simplest comparison possible and the only one that can be anticipated. Nevertheless, when each laboratory "result" is

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[1] This proposed recommended practice is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1953.

[3] For a more complete ASTM publication on the presentation of data and presenting limits of uncertainty of an observed average, see "ASTM Manual on Quality Control of Materials," STP 15-C, January, 1951.

[4] Also see Simon, "Engineers Manual of Statistical Methods," John Wiley and Sons, Inc., New York, N. Y. (1941).

the mean from more than one test, and where the Reproducibility of a method is more than twice as large as the Repeatability, the mean results obtained in the two laboratories may be compared directly with the Reproducibility as described in the following sections without apciable change in confidence level. However, when Repeatability and Reproducibility have the same, or nearly the same, values, then the number of tests constituting a laboratory mean contributes significantly to the confidence in that mean; this situation does not appear to be a very common one.

(*c*) *Estimate of Standard Deviation* is the numerical measure of variability on which the derived terms in the recommended practice is based, and is generally calculated from cooperative tests from which 20 to 30 independent test results are available. The symbol s is used to designate this term as distinguished from the symbol σ associated with a calculation from a much larger sample more representative of the whole population. The value s is calculated[5] as follows:

$$s = \sqrt{\frac{n(X_1^2 + X_2^2 + \dots + X_n^2) - (X_1 + X_2 + \cdots + X_n)^2}{n(n-1)}}$$

where:

$X_1, X_2, \cdots, X_n$ = individual results, and

n = number of test results

Interpretation

3. The following rules for judging the reliability of a set of results are based upon the best available estimate of the standard deviation of the test operation for the given method and are expected to be applicable to the entire population of data developed by strict adherence to that method. These rules are not intended as general rules for the rejection of wild results from very small samples of data obtained by methods other than those standardized on the basis of this Practice.[6]

Repeatability Rejection Rules

4. (*a*) When two results are obtained that differ by less than the Repeatability of the method, report the mean of the two values obtained as the result of the test.

(*b*) When two results are obtained which differ by more than the repeatability of the method, regard one or both of these results as suspect and make two further tests. Multiply the Repeatability of the method by 1.32 (because the range for a sample of 4 is being used instead of the range for 2), and if the difference of the two most divergent results is less than this value, report the mean of all four values as the result of the test.

(*c*) When the difference between the first pair differs by more than the repeatability and that of the second pair does not, and the difference between the two most divergent results exceeds 1.32 times the Repeatability, report the mean of the second pair as the result of the test and discard the first pair of results.

(*d*) When both pairs of results exceed the Repeatability of the method, consider all results as equally suspect and examine the apparatus, procedure, and sample for noncompliance with the conditions prescribed in the method. Correct the conditions, discard previous results, and redetermine a pair of values.

(*e*) Where the number of determinations made exceeds two, for reasons other than unsatisfactory agreement between the first pair of results, the data may still be compared directly with Repeatability to obtain a reasonably satisfactory criterion for rejection. Compare the most suspect result with the mean of the remaining results, and if the difference so obtained exceeds the Repeatability of the method, then that result should be rejected and the mean of the remaining results reported.

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[5] See page 14, footnote 1 of "ASTM Manual on Quality Control of Materials," STP 15-C, January, 1951.

[6] See Blaedel, Meloche, and Ramsay, "A Comparison of Criteria for the Rejection of Measurements," *Journal*, Chem. Ed., Vol. 28, p. 643, 1951, which described method for rejection of a discordant result from a small sample of data (3 to 10 tests), and Dean and Dixon, "Simplified Statistics for Small Number of Observations," *Analytical Chemistry*, Vol. 23, p. 636, 1951.

Repeatability Confidence Limits

5. (*a*) It is important to distinguish between cases in which it is desired to establish both upper and lower limits of the expected mean of an infinite number of results (double limit situation), and those cases in which it is required simply to establish an upper (maximum) limit or a lower (minimum) limit.

(*b*) If one operator working on one apparatus makes a series of n determinations of some quantity obtaining a mean value of $\bar{X}$, then it may be said that the mean of an infinite number of tests by the same operator would fall within certain limits 95 per cent of the time as follows:

$$\text{within } \bar{X} \pm \frac{\text{Repeatability}}{\sqrt{2n}}$$

This allows the gain in precision by one operator to be expected from any increase in the number of results to be evaluated exactly for the confidence level of 95 per cent.

(*c*) If, in obtaining a set of results under conditions of Repeatability, it is required to establish a single limit, either upper or lower, for the expected mean of an infinite number of tests, then the same procedure is followed except that the value given for Repeatability, r, must be multiplied by a factor of 0.84 (Note 3). Thus, it may be said with 95 per cent confidence that the mean of an infinite number of tests by one operator lies either:

$$\text{below } \bar{X} + \frac{0.84r}{\sqrt{2n}}$$

or

$$\text{above } \bar{X} - \frac{0.84r}{\sqrt{2n}}$$

NOTE 3.—To multiply the double limit situation by the factor 0.84 to obtain the single limit situation is not strictly correct since the factor varies slightly according to the precision of the estimate of Repeatability. The magnitude of this variation is, however, quite small, and the use of a single factor is sufficiently accurate for practical purposes.

Reproducibility Suspicion Rule

6. (*a*) If two test results, one reported by each of two laboratories, agree within the limits of Reproducibility, the results (Note 4) should be averaged and assigned as the value for the sample. The mean value of the two laboratory results rather than either one separately should be considered preferable in judging conformance to a limiting specification value.

NOTE 4.—Normally, it is expected that when comparisons between results of two laboratories are made for Reproducibility, single values from each will be compared. If each of the laboratories has produced more than a single result, and if the Reproducibility is more than twice the repeatability, the means of results from each laboratory may be compared as described for single results (see also Note 2).

(*b*) When the values from two laboratories do not agree within the limits of Reproducibility, and one is outside the limiting specification value, then each laboratory should repeat the test on a freshly drawn sample (Note 5). The mean of this second pair of tests should then be observed for agreement as just described.

NOTE 5.—When repeat tests are made, the freshly drawn sample should be equally divided for test by all participating laboratories in order to eliminate the possibility of introducing a gross sampling error into the question of agreement by these tests.

(*c*) If the two laboratories cannot produce a pair of results in agreement with these concepts, it is probable that operations in one of the laboratories is not under control as expected by the method and a third or referee laboratory might be invited to also make the test, again using a freshly drawn sample (Note 5); in this case, multiply the Reproducibility by 1.2 (to convert a range for a sample of two to a range for a sample of three) and compare the two most divergent results with it for acceptance. The average of the three, if acceptance is indicated, is to be considered the best value for the set. Alternatively, tests might be made on a standard or reference sample to demonstrate magnitude of lack of testing control.

TABLE I.—MULTIPLIERS OF THE ESTIMATE OF THE STANDARD DEVIATION FOR CALCULATING THE GREATEST DIFFERENCE BETWEEN TWO RESULTS AT THE 95 PER CENT PROBABILITY LEVEL.

Degrees of Freedom[a]	Multiplier	Degrees of Freedom[a]	Multiplier	Degrees of Freedom[a]	Multiplier
		15	3.01	30	2.89
1	17.97	16	3.00	40	2.86
2	6.08	17	2.98	50	2.84
3	4.08	18	2.97	60	2.83
4	3.92	19	2.96	120	2.80
5	3.64	20	2.95	∞	2.77
6	3.46	21	2.94		
7	3.34	22	2.93		
8	3.26	23	2.93		
9	3.20	24	2.92		
10	3.15	25	2.91		
11	3.11	26	2.91		
12	3.08	27	2.90		
13	3.05	28	2.90		
14	3.03	29	2.89		

[a] Number of separate independent tests minus one ($n - 1$).

Discussion of Repeatability

7. (*a*) For an infinite number of tests by a single operator working with a single apparatus, the standard deviation, σ, is related to the range of a pair of results. In the long run a range of 2.77σ should be exceeded by the operation of random errors only 5 per cent of the time. Since only an estimate of the standard deviation, s_r, is calculated from the limited number of data usually available, this estimate must be multiplied by a factor larger than 2.77 (Table I) for the calculated range to have the same probability level.

(*b*) The criterion for Repeatability in Paragraph (*a*) is built upon the standard deviation of the difference of pairs of results obtained in an individual laboratory and the deviation of this difference from zero. The variance of the difference of two results is twice the variance of individual results, and the standard deviation of the difference is $\sqrt{2}$ (or 1.414) times the standard deviation of the individual results. Although 1.96σ represents the 95 per cent probability limits for the distribution of individual results about the mean, 2.77σ (or $1.414 \times 1.96 \times \sigma$) represents the 95 per cent probability limits for the variation of the difference of pairs from zero. A similar situation applies to Reproducibility.

(*c*) While the definition of Repeatability depends upon the use of a single apparatus, it is not practical to obtain a repeatability standard deviation from a large number of tests by a single operator, using either one or several apparatuses; nor is it reasonable to expect a single operator to be typical for the test in question. Hence, estimates of repeatability standard deviations are obtained by combining (pooling) the differences between results from each of several operators in different laboratories, (carrying out the same determination on identical material) from his mean.

(*d*) When k operators are involved,[7] if Operator 1 makes n_1 determinations of which the mean is $\bar{X}_1$ with any individual result X_1, Operator 2 makes n_2 determinations of which the mean is $\bar{X}_2$ with any individual result X_2, and so on to Operator k, the pooled estimate of the repeatability standard deviation, s_r, can be obtained from:

$$s_r = \sqrt{\frac{\Sigma(X_1 - \bar{X}_1)^2 + \Sigma(X_2 - \bar{X}_2)^2 + \cdots + \Sigma(X_k - \bar{X}_k)^2}{(n_1 - 1) + (n_2 - 1) + \cdots + (n_k - 1)}}$$

When k operators, each from a different laboratory, produce a pair of results with differences of $d_1, d_2 \cdots d_k$, then the above reduces to:[8]

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[7] "Standard Methods for Testing Petroleum and its Products," Institute of Petroleum (London), Appendix E of latest edition.

$$s_r = \sqrt{\frac{d_1^2 + d_2^2 \ldots + d_k^2}{2k}}$$

Discussion of Reproducibility

8. (*a*) The estimate of the reproducibility standard deviation, s_R of the test results from different laboratories is obtained from a limited number of cooperative tests and has a relation to the standard deviation, σ, of the mean of results from each of an infinite number of laboratories as has Repeatability. On the whole, the number of laboratories participating in a given cooperative test program may be less than the number of operators, so that the Reproducibility of a method will be slightly less precisely known than the Repeatability. Continuing the simple case of Section 7(*d*) where k operators, each in different laboratories, produce results the average of which is $\bar{X}_k$ and the grand average is $\bar{\bar{X}}$, then the reproducibility standard deviation, s_R, is calculated from:

$$s_R = \sqrt{\frac{(\bar{X}_1 - \bar{\bar{X}})^2 + (\bar{X}_2 - \bar{\bar{X}})^2 + (\bar{X}_k - \bar{\bar{X}})^2}{k - 1}}$$

(*b*) The value for Reproducibility is obtained by multiplying the calculated value for s_R by the appropriate factor from Table I corresponding to $(k - 1)$ degrees of freedom. It is being assumed here that the mean value produced in each laboratory provides an s_R comparable to that from the first result of each (Note 2). Where this is not the case, then only the first result from each of the k operators in the k laboratories should be used for $\bar{\bar{X}}_k$, and may be calculated as follows:

$$s_R = \sqrt{\frac{\Sigma X^2 - k\bar{\bar{X}}^2}{k - 1}}$$

or

$$\sqrt{\frac{k\Sigma X^2 - (\Sigma X)^2}{k(k - 1)}}$$

Identification

9. This recommended practice is intended to apply when the following or its equivalent appears in the Precision Section of the method:

Precision:

The following data should be used for judging the acceptability of results (95 per cent probability) according to the concept of precision as given in the ASTM Proposed Recommended Practice for Applying Precision Data Given in ASTM Methods of Test for Petroleum Products and Lubricants.

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Range or Sample Description	Repeatability
....	....
....	....

(*b*) The result submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

Range or Sample Description	Reproducibility
....	....
....	....

NOTE 6.—Whenever it is clearly possible to do so, express Repeatability and Reproducibility in the same dimensions as required for reporting results. When the precision is stated as a percentage value, it is intended that the value represent the absolute value of uncertainty of a result also reported as percentage. When the precision is stated as "per cent of the mean," it is intended as a relative measure of uncertainty and may be applied regardless of the units of measurement to be reported by the method.

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[8] Youden, "Statistical Methods for Chemists," John Wiley and Sons, Inc., New York, N. Y., p. 16 (1951).

APPENDIX V

PROPOSED WEATHERING TEST FOR LIQUEFIED PETROLEUM GASES[1, 2, 3]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes a procedure for determining the boiling point of the residue remaining after 95 per cent of a liquid sample of liquefied petroleum gas has been evaporated by weathering.

Outline of Method

2. A 100-ml liquid sample of liquefied petroleum gas, precooled to approximately its boiling point, is collected in a centrifuge tube, and allowed to weather in air as long as its temperature remains below 10 F, or immersed in a 60 to 70 F water bath when the weathering temperature exceeds 10 F. The temperature of the residue is determined when 5 ml of the sample remains. After correction to a barometric pressure of 740 mm of mercury, this temperature is reported as the boiling point of the 5-ml residue.

Apparatus

3. (*a*) *Weathering Tube*, made of heat-resistant glass, and conforming to the requirements shown in Fig. 1.

(*b*) *Tube Support*, for holding the weathering tube by its neck in a vertical position in the water bath.

(*c*) *Water Bath*, made of a shallow container filled with clean water to a depth of $1\frac{1}{2}$ in., and maintained at a temperature of 60 to 70 F.

(*d*) *Thermometer*, ASTM Low Cloud and Pour, having a range of -112 to $+70$ F, and conforming to the requirements for thermometer 6F as prescribed in ASTM Standard E 1.[4]

(*e*) *Precooling Apparatus*, consisting of the following parts, as shown in Fig. 2:

(*1*) *Cooling Vessel.*—Any suitable wide-mouth metal container or Dewar flask, at least $2\frac{1}{2}$ in. in inside diameter, by $11\frac{1}{2}$ in. deep.

(*2*) *Cooling Coil.*—A cooling coil, consisting of approximately 20 ft of $\frac{3}{16}$ in. OD soft copper tubing wound on a hollow mandril at least $2\frac{1}{8}$ in. OD, with adjacent turns touching. The lower end of the coil shall be brought up through the center of the mandril before winding, so that the finished coil will fit snugly the sides of the cooling vessel. When assembled, the top of this coil shall be at least 1 in. below the top of the cooling vessel. The ends of the copper coil shall not extend more than a few inches beyond the mouth of the cooling vessel. The downstream end shall be connected to a $\frac{1}{8}$-in. needle valve provided with an outlet connection not more than 3 in. long.

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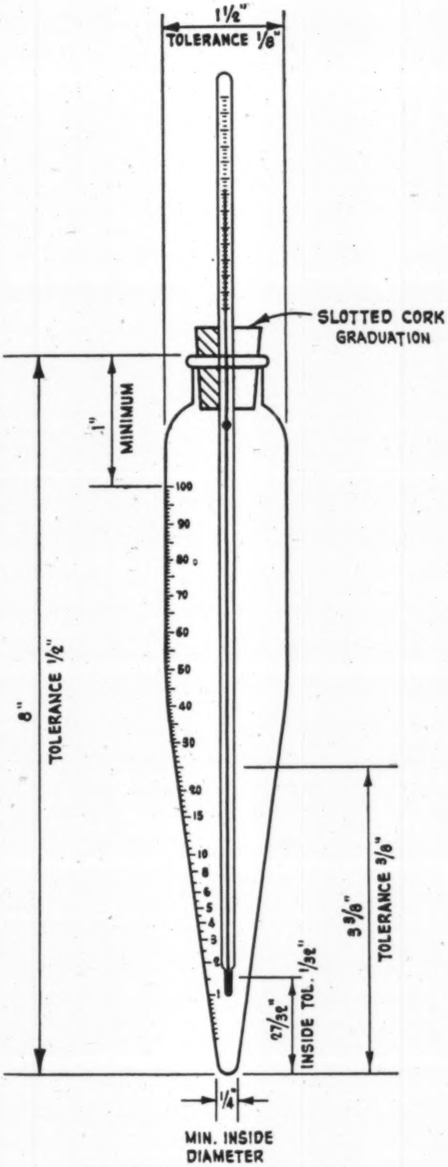
[1] This proposed method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

[2] This proposed method is based on the Natural Gasoline Association of America's LPG Weathering Test, as described in NGAA Publication 2140.

[3] Published as information, June, 1953.

[4] 1952 Book of ASTM Standards, Parts 3, 4, 5.


1½"
TOLERANCE ⅛"
SLOTTED CORK GRADUATION
1"
MINIMUM
8"
TOLERANCE ½"
100
90
80
70
60
50
40
30
20
15
10
8
6
5
4
3
2
1
3⅜"
TOLERANCE ⅜"
27/32"
INSIDE TOL. 1/32"
¼"
MIN. INSIDE DIAMETER

GRADUATION TOLERANCES

Range, ml	Scale Division, ml	Limit of Error, ml
0.0 to 0.1.......	0.05	0.02
0.1 to 0.3.......	0.05	0.03
0.3 to 0.5.......	0.05	0.05
0.5 to 1.0.......	0.1	0.05
1.0 to 3.0.......	0.1	0.1
3.0 to 5.0.......	0.5	0.2
5.0 to 25.0......	1.0	0.5
25.0 to 100.0.....	1.0	1.0

NOTE.—The shape of the lower tip of the tube is especially important. The taper shall be uniform and the bottom shall be rounded as shown in the drawing.

Tubes must be made of heat-resistant glass and thoroughly annealed.

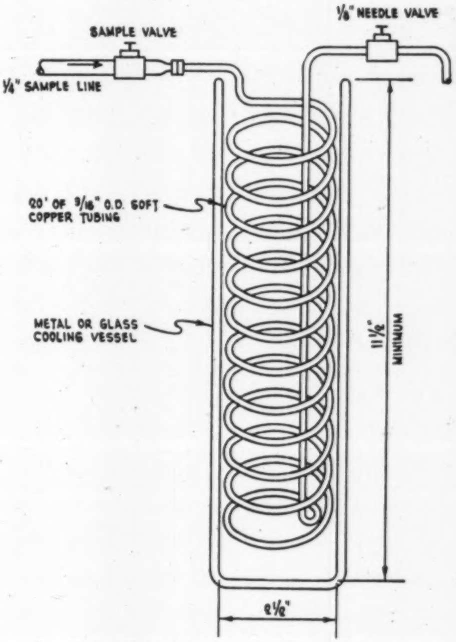
Tubes shall comply in wall thickness to ASTM centrifuge tube requirements.

FIG. 1.—Weathering Tube.

(3) *Precoolant.*—The precooling liquid may be the liquefied petroleum gas from the same container from which the test sample is later to be taken. Other refrigerants having a boiling point lower than the initial boiling point of the sample may also be used. Where safety requires its use, a nonflammable precoolant should be employed.

Sampling

4. Fill the cooling vessel with the precoolant so as to cover the cooling coil, and attach the inlet of the cooling coil to the source of sample with a short-line connection of ¼-in. pipe or larger, and with a sampling valve large enough to prevent vaporization of the material due to drop in pressure across the valve seat. Purge the sampling line and cooling coil


⅛" NEEDLE VALVE
SAMPLE VALVE
¼" SAMPLE LINE
20' OF 3/16" O.D. SOFT COPPER TUBING
METAL OR GLASS COOLING VESSEL
11½"
MINIMUM
2½"

(Coils in drawing extended for clarity.)

FIG. 2.—Precooling Apparatus.

by opening both the sampling valve and the $\frac{1}{8}$-in. needle valve on the downstream end of the cooling coil. After sufficient purging, fill the weathering tube with the sample flowing through the cooling coil. Empty this first sample, add one or two grains of charcoal, and then refill the weathering tube to the 100-ml mark with fresh liquid sample passing through the cooling coil. Carefully insert the thermometer, which has been cooled in the precoolant bath. Center the bottom tip of the thermometer bulb in the tube opposite the 1-ml mark. This may be accomplished by the use of a slotted cork.

Procedure

5. If the temperature of the sample is below 10 F, allow it to weather in the atmosphere until it has reached 10 F. When the temperature of the sample is 10 F or greater, place the weathering tube vertically in the 60 to 70 F water bath to the $1\frac{1}{2}$-ml mark, and allow the contents to weather. Do not remove the thermometer from the weathering tube during the test.

Report

6. With the thermometer in place, note and record the temperature when the liquid level is at 6.0 ml. This corresponds to a hydrocarbon residue of 5.0 ml. Correct the observed temperature to a barometric pressure of 740 mm of mercury by adding 0.6 deg to the observed temperature for each 10 mm the test is conducted *below* 740-mm pressure, or by subtracting 0.6 deg from the observed temperature for each 10 mm the test is conducted *above* 740-mm pressure. Report the corrected temperature as the 5 per cent residue boiling point.

APPENDIX VI

REPORT ON PROPOSED METHODS FOR ANALYSIS OF GRAPHITE UNDER CONSIDERATION BY RESEARCH DIVISION XII ON GRAPHITE TESTS

This report is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

A survey of existing methods of analysis of graphite shows a wide variety of methods. Many of them apparently are based on accepted methods of coal analysis because not only is a method for "volatile" given but the final results are dependent on this determination. Lubricating grades of graphite are very low in volatile and, furthermore, volatile has little or no significance.

Section B of Research Division XII of Committee D-2 is working on methods that will not only be significant for lubricating grades of graphite but for most other graphites as well.

The method for moisture is widely-used. The term "moisture and other volatile matter" is included to satisfy the fact that in heating at 105 C the loss is not rigidly limited to water. The method is capable of greater precision than 0.1 per cent, but this figure was deemed adequate.

With graphites, the nongraphitic portion is of more significance than an exact determination of graphitic carbon (pure graphite). The practice has been, however, to report graphitic carbon. Bowing to custom, therefore, there has been included a calculation for graphitic carbon.

When graphitic carbon was calculated from determinations of volatile and ash, a wide discrepancy between laboratories (and sometimes within laboratories) was noted. The error was pinned down to oxidation of the graphite in the heating process to determine volatile. Thus, not only was the volatile in error but, more important, the graphitic carbon as well.

It was evident that a determination of ash alone was insufficient, because it would not show combustible adulterants. Practically, the only adulterant not affecting the ash is amorphous carbon. Amorphous carbon can be separated from graphite by a heavy liquid that partitions their respective densities. Ethylene bromide was chosen because it has a density between carbon and graphite and because it may be used as purchased, without adjustment.

On the dry basis, a precise analysis of graphite can be made by determining the amorphous carbon and the ash. The methods given herewith are simple, reproducible and significant. Methods of greater precision, such as the combustion train for total carbon, have been considered and tested. They may appear later as alternate methods.

Cooperative tests now under way in Research Division XII have demonstrated that the methods given here are better suited to the laboratories of sellers and consumers of graphite. These proposed methods, developed by Section B on Chemical Analysis, are as follows.

PROPOSED METHODS FOR ANALYSIS OF GRAPHITE

Moisture

Procedure

1. Transfer 5 g of the sample, weighed to 0.001 g, to a wide-mouth weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr at 105 ± 2 C, insert the stopper, cool, and weigh.

Calculation and Report

2. (*a*) Calculate the percentage moisture and other volatile matter as follows:

Moisture and other volatile matter, per cent

$$= \frac{\text{wt loss on heating}}{\text{wt of sample}} \times 100$$

(*b*) Report to the nearest 0.1 per cent.

Carbon (Amorphous)

Procedure

3. (*a*) Rub up approximately 5 g of the dried sample in a mortar with a pestle and weigh 1 g, to the nearest 0.001 g, into a 15-ml cone-shape centrifuge tube. Add 10 to 15 ml of ethylene bromide (sp gr 2.18 to 2.19) and shake well by hand. Centrifuge in excess of 1200 rpm for a minimum of 20 min; then remove the centrifuge tube and allow to stand undisturbed for 1 hr. There should be a clear line of demarcation between the liquid and the solids.

(*b*) If any material has collected at the top of the liquid, it shall be reported as carbon. Transfer the floating solids to a tared Gooch crucible and wash with acetone; dry at 105 ± 2 C to constant weight.

(*c*) Because occluded air may cause graphite to float, giving erroneous results, the carbon found may exceed specification limits. When limits are exceeded, the test shall be repeated on a sample ground to pass a No. 100 (149-micron) sieve.[1]

Calculation and Report

4. (*a*) Calculate the carbon as follows:

Carbon, per cent =

$$\frac{\text{wt gain in Gooch crucible}}{\text{wt of sample}} \times 100$$

(*b*) Report to the nearest 0.1 per cent.

Ash

Procedure

5. Weigh into a shallow, previously ignited and weighed, porcelain or platinum dish or boat (Note 1) 1 g of the dried sample, and spread thinly and evenly over the entire bottom. Heat in a muffle with free access of air at 950 ± 25 C until all combustible matter is consumed (Note 2). Cool the vessel in a desiccator, and weigh.

Calculation and Report

6 (*a*) Calculate the ash as follows:

Ash, per cent =

$$\frac{\text{wt of sample after ignition}}{\text{wt of sample}} \times 100$$

(*b*) Report to the nearest 0.1 per cent.

Graphitic Carbon

Calculation and Report

7. (*a*) Calculate the graphitic carbon as follows:

Graphitic carbon, per cent
$$= 100 - (\text{per cent ash} + \text{per cent carbon})$$

(*b*) Report to the nearest 0.1 per cent.

NOTE 1.—For referee purposes, use a flat-bottom dish having a diameter of 49 mm.

NOTE 2.—Three hours is usually enough time for consumption of the combustable matter.

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[1] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (ASTM Designation: E 11), 1952 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

A joint meeting of Committee D-3 on Gaseous Fuels and its seven subcommittees was held in State College, Pa. on July 18, 1952. Four of the subcommittees also held individual meetings on the same day. Two additional subcommittee meetings were held in Detroit, Mich., on March 2, 1953.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Sampling Manufactured Gas which had been prepared by Subcommittee I. The method was accepted by the Standards Committee on December 12, 1952, and it appears in the 1952 Book of ASTM Standards, Part 5, bearing the designation D 1247 - 52 T.

NEW TENTATIVE

Committee D-3 recommends the Method of Sampling Liquified Petroleum Gases for publication as tentative as appended hereto.[1] This method was developed jointly in cooperation with Committee D-2 on Petroleum Products and Lubricants. In its original form the method was published in 1950 as information, and in 1952 with some minor revisions was incorporated in the Tentative Method of Sampling Petroleum and Petroleum Products (D 270 - 52 T).[2] Committee D-3 believes that this sampling method should be published separately under an individual ASTM designation. Committee D-2 concurs in this proposal and the method appended hereto[1] is accordingly recommended jointly by both committees.

ADOPTION OF TENTATIVES AS STANDARDS

Committee D-3 recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of:

Analysis of Natural Gases by the Volumetric-Chemical Method (D 1136 - 50 T),

Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 - 50 T),

Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142 - 50 T), and

Sampling Natural Gas (D 1145 - 50 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

AMERICAN STANDARDS

Approval as American Standard was accorded by the American Standards

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement of Book of ASTM Standards, Part 5.

[2] 1952 Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Association to the ASTM Standard Method of Test for Calorific Value of Gaseous Fuels (D 900 - 48 T). This was approved under the designation ASA Z68.1–1953.

Following its adoption as standard by the Society, the ASTM Method of Test for Specific Gravity of Gaseous Fuels (D 1070 - 52) was also submitted to the American Standards Association for approval as American Standard as recommended by Committee D-3. Approval was accorded under the designation ASA Z69.1–1953.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Collection of Gaseous Samples (F. E. Vandaveer, chairman).—This subcommittee originally prepared the Method of Sampling Liquified Petroleum Gases which is now recommended for publication as tentative, as mentioned earlier in this report.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—Main attention has been devoted to a revision of the Tentative Method for Measurement of Gaseous Fuel Samples (D 1071 - 49 T) to clarify some meter calibration details not in full agreement with the Standard Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 - 48). Decision was reached at the July 18 meeting of the subcommittee to appoint a task force to prepare these revisions. It was also agreed that any resulting revisions would be cleared through Subcommittee III. The report of the task force is now being awaited.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. S. Jessup, chairman).—Proposals offered for revision of the Standard Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 - 48) were considered at the March 2 meeting. Principal features related to calibration of the $\frac{1}{10}$ cu ft wet test meter, reproducibility of results, and summarized operating instructions. It was agreed that recommendations of the task force now studying wet test meter calibration should be secured before proceeding with any revisions on the present procedure. The chairman has circulated to all members a request for additional information on reproducibility of results when employing the method as written, and also requesting letter ballot approval of proposed summarized operating instructions for inclusion in an appendix to the method.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (L. T. Bissey, chairman).—Attention has been concentrated on methods for determination of total sulfur, following the plan adopted some time ago. Agreement was reached at the July 18 meeting to proceed with preparation of a revised referee method for presentation to the members, so that definite decision could be made on developing it as a tentative or withdrawing it from further consideration. A revised method prepared by a working group has been distributed to members for letter ballot approval.

Preparation of the Wilson-Kemp lamp method for determination of total sulfur is now under way. Study is also being devoted to other methods for total sulfur determination with special attention to the precision and application of each.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (S. W. Burdick, chairman).—The subcommittee is conducting cooperative work on Brickell's method for water vapor in gas which consists essentially of scrubbing water vapor out of the gas with anhydrous ethylene glycol, with subsequent titration of the glycol-water mixture with Karl Fischer reagent.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—A procedure for analysis of carbureted water gas by the mass spectrometer has been developed by a group consisting of several members of the subcommittee and distributed for study and comment. It is proposed to revise the test to include any changes which may be necessary as the result of comments received. The method will then be presented to Committee D-3 and the entire subcommittee for approval.

This report has been submitted to letter ballot of the committee, which consists of 38 members; 31 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

K. R. KNAPP,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards a proposed Tentative Method of Analysis of Carbureted Water Gas by the Mass Spectrometer. The method was accepted by the Standards Committee on November 9, 1953, and it appears in the 1953 Supplement to Book of ASTM Standards, Part 5, bearing the designation D 1302 – 53 T.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the past year: in New York, N. Y., on June 25, 1952, and in Detroit, Mich., on March 5, 1953.

In the period since preparation of the last annual report, Committee D-4 has lost four of its members by death. It is with sincere regret that the committee records the passing of these gentlemen.

Lawrence Ortolani, Materials and Tests Engineer of the Texas Highway Department, died April 25, 1952. He had been a committee member representing that highway department since 1950.

Edward Henry Berger, Consulting Chemist, who became a personal member of ASTM in 1934, had represented the Johns-Manville Corp. in Committee D-4 since 1934. His death occurred on May 12, 1952.

Claude Leon McKesson, Research Consultant, became a member of ASTM in 1930 and a member of Committee D-4 in 1931. Mr. McKesson died on July 21, 1952.

John Strother Miller, Asphalt Technologist affiliated with Miller-Warden Associates, passed away on August 23, 1952. He became a member of the Society in 1903 and was elected to Honorary Membership in 1946. He had been a member of Committee D-4 since 1914.

Dr. Berger, Mr. McKesson, and Mr. Miller not only served actively on Committee D-4 over a long period of years, but also participated in various other activities of the Society and made many important contributions to its progress. In recognition of their high attainments and the loss which members of Committee D-4 feel in their passing, appropriate memorial resolutions were adopted by the committee and have been entered in the permanent records of Committee D-4 and of the Society.

At its meeting in June, 1952, the committee recognized the long service of four of its members by electing them to honorary membership:

W. H. Fulweiler, a member of ASTM since 1909, and a past President and Honorary Member of the Society, has been a member of Committee D-4 since 1910.

H. S. Mattimore, a member of ASTM since 1914, served Committee D-4 as chairman from 1924 to 1926, and was a recipient of the Award of Merit at the Fiftieth Anniversary Meeting of the Society.

H. B. Pullar has been a personal member of the Society since 1923, but in 1910 became a member of Committee D-4 as representative of the American Asphaltum and Rubber Co.

Herbert Spencer became an ASTM member and a member of Committee D-4 in 1909.

All of these have served continuously as members of Committee D-4 and have contributed greatly to its progress, particuarly in those years when the technology of road materials was in its earliest stages of development and many of the practical principles of road materials control as we use them today were established.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-4 presented to the Society, through the Administrative Committee on Standards, recommendations for publication of the following:

Tentative Revision of Standard Methods of Testing:

Emulsified Asphalts (D 244 - 49), and

Tentative Specifications for:

Materials for Soil-Aggregate Subbase, Base, and Surface Courses (D 1241 - 52 T).

These recommendations were accepted by the Administrative Committee on Standards on September 5, 1952, and appear in the 1952 Book of ASTM Standards, Part 3. The tentative revision of D 244 - 49 provides a method for the control of consistency of Type RS-2 emulsified asphalts by use of a test for Saybolt-Furol viscosity at 50 C, as required by the tentative revision of Standard Specifications for Emulsified Asphalt (D 977 - 49), issued June, 1951.

The Tentative Specifications D 1241 - 52 T are under the joint jurisdiction of Committee D-4 and Committee D-18 on Soils for Engineering Purposes and have been developed by these committees and by the American Association of State Highway Officials to put requirements for soil-aggregate highway materials on a more logical and systematic basis. These specifications cover a wide range of materials suitable for the stated uses, and recognize variations necessary to meet local conditions of service and availability of materials. In effect, they replace Tentative Specifications for Materials for Stabilized Base Course (D 556 - 40 T) and Materials for Stabilized Surface Course (D 557 - 40 T) which are recommended subsequently in this report to be withdrawn.

TENTATIVE REVISIONS OF STANDARD

Standard Specifications for Bituminous Mixing Plant Requirements (**D 995 - 51**).[1] —To provide for adequate control when aggregates are blended, the committee believes that additional requirements on the feeder for the drier are desirable, and recommends publication of the following tentative revision of this standard:

Section 2(c).—Add the following to this paragraph:

> When aggregates must be blended at the cold elevator to meet the requirements of the job mix tolerances, a mechanically interlocked feeder of two or more bins shall be provided.

The portion of these specifications relating to continuous mixing plants does not clearly specify the mixing time for this type of plant. The committee recommends, therefore, publication of a tentative revision of the standard, as follows:

Section 4(d).—Add the following to this paragraph:

> The weight per unit volume relationship of the coated loose mix shall be determined and, by reference to the volume gage on the side of the mixer, the dead weight at any operating height and the mixing time shall be calculated, using the following formula:

$$\text{Mixing time in seconds} = \frac{\text{Pugmill dead capacity in pounds}}{\text{Pugmill output in pounds per second}}$$

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee D-4 joins with Committee C-9 on Concrete and Concrete Aggregates in recommending approval of the following revision in the *Standard Methods of Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine* (**D 289 – 46**)[1] for immediate adoption, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

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[1] 1952 Book of ASTM Standards, Part 3.

Section 5(a).—Add the following to the tabulation of gradings:

Grading	Passing Sieve	Retained on Sieve	Percentage of Sample
E	⅜ in.	No. 4	50
	½ in.	⅜ in.	50

The purpose of this recommendation is to make the gradings specified in the ASTM method of test correspond to those provided in the similar method of the American Association of State Highway Officials.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-4 recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for Preformed Expansion Joint Filler for Concrete, Bituminous Type (D 994 – 48 T), without revision, and

Tentative Method of Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 – 51 T), editorially revised as follows:

Section 4(e).—Change the first sentence of this paragraph, by addition of the italicized words, to read "In case the specimen has been obtained from a pavement and contains moisture, it is necessary to correct weights 'A', '*D*', *and* '*E*' for the weight of moisture."

Change the last line of this paragraph, by addition of the italicized words, to read "and apply the appropriate correction to weights A, *D*, *and E*."

Section 5.—Insert the word "dry" before "specimen" in definitions of the terms "D" and "E."

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Specifications for Granite Block for Pavements (**D 59 – 39**)[1].—Committee D-4 recommends that the tentative revision of this standard, issued September 12, 1951, be approved for reference to letter ballot of the Society for adoption as standard.

Standard Specifications for Emulsified Asphalt (**D 977 – 49**)[1].—Committee D-4 recommends that the tentative revision of this standard, issued June, 1951, be approved for reference to letter ballot of the Society for adoption as standard.

Standard Specifications for Bituminous Mixing Plant Requirements (**D 995 – 51**).[1]—Committee D-4 recommends that the tentative revision of this standard, issued June, 1952, be approved for reference to letter ballot of the Society for adoption as standard.

WITHDRAWAL OF TENTATIVES

In view of the publication of the Tentative Specifications for Materials for Soil-Aggregate Subbase, Base, and Surface Courses (D 1241 – 52 T),[1] which were accepted on September 5, 1952, Committee D-4 recommends the withdrawal of the Tentative Specifications for Materials for Stabilized Base Course (D 556 – 40 T) and for Materials for Stabilized Surface Course (D 557 – 40 T).

TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-4 and the joint subcommittee of Committees D-4 and D-18 have reviewed existing tentative specifications and methods of test which have been published by the Society for two years or more without revision, and Committee D-4 recommends that the following be continued as tentative without revision:

Tentative Specifications for:

Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 51 T).—These specifications should remain tentative for at least another year to develop suggestions for improvement of their requirements.

Asphalt Cement for Use in Pavement Construction (D 946 - 47 T).—These specifications should be retained as tentative pending the development of additional requirements to secure improved control of the properties and uniformity of asphalt cements.

Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 51 T).—Although no criticism of these specifications has been received, the committee believes that they should be continued as tentative for at least another year to develop suggestions for modification of the requirements.

Tentative Methods of Test for:

Soft Particles in Coarse Aggregates (C 235 - 49 T).—The committee recommends continuing this method as tentative for further consideration of its requirements by Committees D-4 and C-9.

Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T).—This tentative method has been in extensive use for many years, and should be retained. It does not have the reproducibility believed desirable for recommending adoption as standard, and the principal reason for inadequate reproducibility has been established as poor control of ventilation in the ovens used for the test. The need for adequate requirements applicable to test ovens has been brought to the attention of Committee E-1 on Methods of Testing, which is formulating specifications to control the performance of ovens used for heating tests.

Moisture-Density Relations of Soils (D 698 - 42 T).—Committee D-18 on Soils for Engineering Purposes, which has joint jurisdiction with Committee D-4 on this tentative method, is considering revision of the method.

Soil-Bituminous Mixtures (D 915 - 47 T).—Further study of this procedure is desirable in respect to correlation with construction control and service performance of highway soils. The method is in use by a number of highway construction agencies, and it is expected that a subcommittee of Committee D-18, which has joint jurisdiction of this tentative with Committee D-4, will assemble appropriate data on the applicability and utility of the tentative method under practical conditions.

Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (D 916 - 47 T).—Considerable information has been obtained by use of this apparatus, and it is expected that, when the data are correlated with the service behavior of the soils, a revision of the method may be indicated. Further research on the utility and application of this method of testing appears desirable.

Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 - 49 T).—In view of work in progress on tests of compacted bituminous mixtures that may affect this tentative method, it should be continued as tentative for at least another year.

Bitumen Content of Paving Mixtures by Centrifuge (D 1097 - 50 T).—A study is in progress on the application of this method to bituminous paving mixtures containing aggregate of larger maximum size than is described in the scope of this tentative method. This study may lead to recommendations for revision of the method, which should remain tentative pending conclusion of the investigation.

Tentative Revision of Standard:

Definitions of Terms Relating to Materials for Roads and Pavements (D 8 - 52).—The committee is considering further revision of the tentative definition for "tar" issued in June, 1949.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) is continuing its cooperation with the Triaxial Institute, which is engaged in fundamental studies of triaxial testing and at present is concentrating on an investigation of methods for the compaction of specimens, a subject of importance in all physical tests for compressed bituminous mixtures. Several studies bearing on the influence of various components on the properties of mixtures have been made and are continuing under the auspices of the subcommittee; these include a series of tests to evaluate the effect of viscosity of asphaltic binders and another series of tests involving emulsified asphalts. The subcommittee has prepared a proposed tentative method of test for stability and flow value of bituminous mixtures by the Marshall test, and is considering information on the Hveem stabilometer and cohesiometer procedures with a view to preparation of proposed tentative methods.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee B-5 on Softening Point (I. E. Manning, chairman) has made several series of cooperative tests in connection with the effort to prepare a method of test having wide application in the determination of softening point by the ring and ball apparatus, replacing Standard Method of Test D 36 – 26 and Tentative Method of Test E 28 – 51 T. It is indicated that a satisfactory method can be developed.

Subcommittee B-7 on Viscosity and Float Tests (C. A. Benning, chairman) has completed cooperative tests to obtain data on the reproducibility of a proposed tentative method for Engler specific viscosity, and is analyzing the data for preparation of a statement on reproducibility to be incorporated in the method.

Subcommittee B-12 on Structural Properties of Mineral Aggregates (Stanton Walker, chairman) has prepared a proposed tentative method of test for void content of manufactured sand, which has been distributed to the committee with a view to probable consideration as a new tentative method. This proposed method provides for determination of voids on a specified grading and is expected to furnish information concerning characteristic shape of particle. Other procedures for measuring shape of particles are also under consideration. Another activity of the subcommittee is a study of procedures for evaluating the contribution which different aggregates might make to the slipperiness of pavement surfaces.

Subcommittee B-14 on Specific Gravity of Fine and Coarse Aggregates (H. T. Williams, chairman) has been investigating specific gravity tests to determine the relationship between values obtained by various procedures, with the objective of establishing a significant method for application to mixtures with bituminous or other binders. It is indicated that further research is necessary.

Subcommittee B-15 on Sampling Road Materials (C. E. Proudley, chairman) is making a study of sampling bituminous mixtures, with a view to improvement in the requirements of Standard Methods D 979 – 51.

Subcommittee B-16 on Setting Qualities of Bituminous Materials (A. B. Cornthwaite, chairman) has completed several extensive series of cooperative tests to develop a method to measure the curing properties of asphaltic cut-back materials, and has planned further work on this difficult problem.

Subcommittee B-25 on Effect of Water on Compressed Bituminous Mixtures (C. A. Carpenter, chairman) plans to study the behavior of mixtures containing porous aggregates, some of which have shown an increase in strength after immersion in tests by Tentative Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 – 49 T).

Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates (C. M. Hewett, chairman) has continued to investigate possible methods of evaluating the performance of bituminous coatings on aggregates in the presence of water. During the past year the application of a radioactive tracer technique to the measurement of "stripping" has been studied and has been shown to warrant further investigation. A photoelectric reflectance method for measuring the degree of "stripping" has also been tried, determining its limitations for use with varying types of aggregate.

Subcommittee C-4 on Specifications for Emulsified Asphalts (F. H. Baumann, chairman) is considering the desirability of revision of the standard specifications for emulsified asphalt with a view to reduction in number of types and simplification of the differentiation between types.

Subcommittee C-12 on Specifications for Bituminous Surface Treatments (W. H. Mills, chairman) has prepared proposed specifications to cover aggregates for use in single and multiple surface treatments and also is formulating a recommended practice on use of materials in surface treatment. Both of these are to receive thorough study before a recommendation is presented for their approval.

Subcommittee D-3 on Expansion Joint Materials (D. O. Woolf, chairman) is critically reviewing the requirements of Tentative Specifications for Preformed Expansion Joint Fillers for Concrete, Nonextruding and Resilient Types (D 544 – 52 T), considering several suggestions for modification presented during the past year. A program of cooperative tests has been outlined to obtain data on some of the suggested changes.

RESEARCH ACTIVITIES

In annual reports for the past four years, Committee D-4 has included a general summary of the more important research activities of its subcommittees. While the investigations in progress have not resulted in recommendations in the 1953 Annual Report for approval of additional tentative methods and specifications, continuing progress in development of new methods of testing is demonstrated in the preceding review of subcommittee activities. Of particular interest are the various investigations of methods for testing bituminous mixtures and evaluating their probable performance under service conditions under way by Subcommittees B-2, B-25 and B-26; applications of radioactive and photoelectric techniques to a difficult problem of measurement are exemplified in the studies conducted by Subcommittee B-26. The problems which Subcommittees B-12 and B-16 have undertaken should also be mentioned as among those which are especially difficult of solution, requiring new methods of approach and extended research

This report has been submitted to letter ballot of the committee, which consists of 143 members; 109 members returned their ballots, of whom 104 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. A. CARPENTER,
Chairman.

B. A. ANDERTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of:

Standard Definition of Terms Relating to Materials for Road and Pavements (D 8 – 52),
Standard Method of Test for Distillation of Tars and Tar Products (D 20 – 52), and
Standard Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 – 49).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the tentative revisions appear in the 1953 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE*

Committee D-5 on Coal and Coke held meetings in Atlantic City, N. J., on June 25, 1952; and in Detroit, Mich., on March 2, 1953. The Advisory Subcommittee held meetings in Chicago, Ill., on September 5, 1952; and in Detroit, Mich., on March 2, 1953.

During the year five members were added to the committee, five members resigned, and two members died, resulting in a total membership of 63, of whom 19 are classified as consumers, 18 as producers, and 26 as general interest members. For voting purposes, the 19 consumer members have 18 votes, the 18 producer members have 17 votes, and the 26 general interest members have 24 votes, resulting in a total voting membership of 59.

At the Detroit meeting, Committee D-5 decided to participate actively in the work of the International Organization for Standardization (ISO) Technical Committee 27 on Solid Mineral Fuels, and a subcommittee is being formed to implement such participation.

R. F. Abernethy, chairman of Section A, Subcommittee XXI on Methods of Analysis, attended a meeting of ISO/TC 27 Working Group on Volatile Matter Determination during the week of April 13, 1953, at the Fuel Research Station, London, England. At this meeting representatives of the participating countries demonstrated their national methods of volatile matter determination with apparatus brought from their own countries. Mr. Abernethy contributed, for comparative testing, several samples of American coals.

W. M. Bertholf, chairman of the Mechanical Sampling Section of Subcommittee XIII on Coal Sampling, has prepared a paper on current ASTM work on coal sampling for presentation at the next ISO/TC 27 committee meeting to be held October 5 to 9, 1953, at the British Standards Institution, London, England.

W. A. Selvig, Committee D-5 Liaison Officer to ISO/TC 27 and the Working Party on Classification, Coal Committee, Economic Commission for Europe, attended a meeting of the Classification Working Party in Geneva, Switzerland, on July 22 and 23, 1952.

ADOPTION OF TENTATIVE AS STANDARD

Committee D-5 recommends that the Tentative Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances (**D 980 - 48 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard. This recommendation has been submitted to letter ballot of the committee; 57 members returned their ballots, of whom 34 consumer and general interest members voted affirmatively; 16 producer members voted affirmatively; one consumer member voted negatively; and five consumer and general interest members and one producer member reported "not voting."

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 5.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman) has agreed to postpone the projected Symposium on Coal Sampling from June, 1953, to the ASTM Annual Meeting in June, 1954. This decision was reached because of the need for more time to complete experiments and the writing of several papers. During the year the subcommittee also approved by letter ballot the recommendation that Tentative Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances (D 980–48 T) be adopted as standard.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman) is resubmitting to letter ballot the proposed revision of the Standard Method of Test for Free Swelling Index of Coal (D 720–46), which would permit measurement of odd-shaped buttons by an equivalent area method. The subcommittee is also continuing its investigations of the Gieseler plastometer, and agreed to undertake a new assignment to study the Gray-King and Arnu laboratory coking tests in connection with their use for classifying coals by rank.

Subcommittee XVIII on Classification of Coals (H. J. Rose, chairman) has agreed to undertake a new assignment to study a proposed international scheme for classification of coals by rank, which method is advocated by the ECE Working Party on Classification. The subcommittee requested the aid of Subcommittee XV on Plasticity and Swelling of Coal to evaluate small-scale laboratory coking tests, such as the Gray-King and Arnu dilatometer tests.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (J. B. Romer, chairman) was assigned the task of studying the ASME Power Code procedure and reporting its suitability for an ASTM method.

Subcommittee XXI on Methods of Analysis (O. W. Rees, chairman).—Section A of the subcommittee, which has been assigned the Proximate Analysis, has been working on a method for determination of inherent moisture based on an equilibration procedure in line with the following definition prepared by Subcommittee II on Nomenclature and Definitions: "*Inherent moisture* of coal is the moisture content retained after removal of extraneous moisture by equilibration at 30 C and 97 per cent humidity." Section A has also been working actively on the procedure for volatile matter determination, and has decided that the present modified procedure may need revision.

Section B on Ultimate Analysis has completed the seventh proposed revision of procedures for carbon and hydrogen determinations, and hopes to recommend these methods as standard to Subcommittee XXI at the Atlantic City meeting in June, 1953. Revisions of the procedures for oxygen and nitrogen determinations are being considered, and the nitrogen method is well along toward completion.

Section C on Miscellaneous Analysis has undertaken the study of the fusibility of ash determination as its first task.

Subcommittee XXII on Physical Tests of Coke (B. P. Mulcahy, chairman) has been assigned the task of reviewing completely all ASTM coke test procedures; and the report of the Coke Evaluation Project, sponsored by the American Iron and Steel Institute and the American Coke and Coal Chemicals Institute, is being used as a foundation for its deliberations. This report has been supplemented by a questionnaire to summarize the opinions of the subcommittee regarding present ASTM tests on coke. In addition, several coke plants have agreed to collect data on a coke evaluation test program covering a year's operation. A section was appointed to study multiple correlation procedures to attempt to calculate weight per cubic foot from the screen test and apparent specific gravity.

Subcommittee XI on Coal Friability and Subcommittee XVII on Significance of Tests of Coal and Coke were discharged.

This report has been submitted to letter ballot of the committee, which consists of 59 voting members; 51 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. W. ANDERSON,
Chairman.

O. P. BRYSCH,
Secretary.

REPORT OF COMMITTEE D-6
ON
PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 25, 1952, and on February 20, 1953, both in New York City. The Advisory and other subcommittees held meetings on the same dates.

At the present time, Committee D-6 consists of 87 members, of whom 73 are voting members; 30 are classified as producers, 26 as consumers, and 31 as general interest members.

In order to improve the operations of the committee and to expedite the work, the subcommittee structure is being reorganized. It is presently proposed to split up the existing Subcommittee I on Paper Testing Methods into three subcommittees.

The committee has gone on record as favoring a symposium on Tension Testing of Nonmetallic Sheet Materials at the 1954 Annual Meeting as a means towards correlating existing methods and developing new methods. It is felt that there should be standardization of tensile testing methods by several of the technical committees.

NEW TENTATIVE

The committee recommends that the following method be accepted for publication as tentative, as appended hereto:[1]

Tentative Method of Test for:

Dimensional Changes of Paper with Changes in Moisture Conditions.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following three tentatives be approved without revision for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Water Soluble Matter in Paper (D 1162 – 51 T),[2]
Lint of Paper Towels (D 1163 – 51 T),[2] and
Ring Crush of Paperboard (D 1164 – 51 T).[2]

TENTATIVE REVISION OF STANDARD

The committee recommends a tentative revision of the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (**D 644 – 44**) in the form of the completely revised tentative method appended hereto.[3] It is planned that this complete revision will replace the existing standard when it is adopted as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following six tentative methods which have stood for two years without revision be continued as tentative:

Tentative Method of Test for:

Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 – 44 T),
Analytical Filter Papers (D 981 – 51 T),
Stretch of Paper and Paper Products under Tension (D 987 – 48 T),
Water Vapor Permeability of Paper and Paperboard (D 988 – 51 T),
Fiber Analysis of Paper and Paperboard (D 1030 – 49 T), and

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

[2] 1952 Book of ASTM Standards, Part 7.

[3] The tentative revision, in the form of a new tentative, was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

Chloride Content of Paper and Paper Products (D 1161 - 51 T).

The recommendations appearing in the report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

Activities of Subcommittees

Subcommittee I on Paper Testing Methods (R. H. Lace, chairman) has been active in developing new methods of test, and has also continued its critical study of various methods of test for paper and paper products that have been adopted by other agencies such as the Technical Association of the Pulp and Paper Industry. The subcommittee prepared the new test method which is recommended for publication as tentative.

A tentative revision of the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (D 644 - 44) is submitted with this report in the form of a completely revised tentative method.

Work is being done which may lead to proposed revisions in the Standard Method of Conditioning Paper and Paper Products for Testing (D 685 - 44), Bursting Strength of Paper (D 744 - 46), Wet Tensile Breaking Strength of Paper and Paper Products (D 829 - 48), Resistance of Paper to Passage of Air (D 726 - 48), Turpentine Test for Grease Resistance of Paper (D 722 - 45), Sampling Paper and Paper Products (D 585 - 42), Basis Weight of Paper and Paper Products (D 646 - 5)), Tensile Breaking Strength of Paper and Paper Products (D 828 - 48), Mineral Filler and Mineral Coating of Paper (D 686 - 48), Wire and Felt Sides of Paper (D 725 - 45), and Fiber Analysis of Paper and Paperboard (D 1030 - 49 T). A possible modification of the method for stretch (D 987) to adapt it to creped paper is under study.

The proposed method of test for Erasing Quality of Paper was published for information purposes in the October issue of the ASTM Bulletin.

Subcommittee IV on Container Board (W. B. Lincoln, Jr., chairman) devoted most of its efforts this year to extensive round-robin tests which will result in proposed revision of Method D 781 - 44 T. Studies in cooperation with Subcommittee I are being made which may result in proposed revision of the Standard Method of Sampling Paper and Paper Products (D 585 - 42).

This report has been submitted to letter ballot of the committee, which consists of 73 voting members; 46 members returned their ballots, of whom 43 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. R. Willets,
Chairman.

R. H. Carter,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-7

ON

WOOD*

Committee D-7 on Wood met in Chicago, Ill., on March 16–17, 1953. The following subcommittee meetings preceded the meeting of the main committee: Subcommittee I on Specifications for Timber, Subcommittee VI on Timber Preservatives, Subcommittee VII on Wood Poles and Cross Arms, Subcommittee VIII on Modified Wood and Wood-Base Materials, and Subcommittee XV on Structural Fiberboards.

Nine applicants were elected to membership on the committee during the year.

NEW TENTATIVES

The committee recommends that the following new specifications and methods of chemical analysis as appended hereto[1] be accepted for publication as tentatives:

Tentative Specifications for:

Copperized Chromated Zinc Chloride, and
Pentachlorophenol.

Tentative Methods of:

Chemical Analysis of Copperized Chromated Zinc Chloride, and
Chemical Analysis of Pentachlorophenol.

ADOPTION OF TENTATIVE AS STANDARD

The Committee recommends that the Tentative Methods of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (**D 1101 - 50 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF TENTATIVE

The committee recommends that the following revisions of the Tentative Specification for Round Timber Piles (**D 25 - 52 T**)[2] be approved and that the specification be continued as tentative:

Section 4 (*a*).—Fourth sentence, add "Southern pine" at the beginning of the sentence, preceding "Piles."

Section 6.—Change heading from "Circumference" to "Circumferences and Diameters." Renumber this section as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows: "(*b*) The ratio of the maximum to the minimum diameter at the butt of any pile shall not exceed 1.2."

Section 9 (*b*).—Revise to read as follows: "(*b*) All knots and limbs shall be trimmed or smoothly cut flush with the surface of the pile, except that knots may be hand-trimmed flush with the surface of the swell surrounding the knot."

NOTE 9.—Delete.

Section 10 (*a*).—Revise to read as follows: "(*a*) Piles are classified according to the extent of bark removal as clean-peeled, rough-peeled, or unpeeled."

NOTE 10.—Delete.

Section 10 (*b*).—Revise to read as follows: "(*b*) Clean-peeled piles require the removal of all outer bark. In addition, at least 80 per cent of the inner bark, well distributed over the surface of the pile, shall be removed. For proper preservative treatment, no strips of inner bark wider than ½ in. shall remain."

Table I.—Change minimum diameters 3 ft from butt, Class A piles, Douglas-fir, etc., under 40 ft, oak, etc. under 30 ft, cedar under 30 ft, from 13 in. to 14 in. Change corresponding circumferences from 41 in. to 44 in.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 4.

[2] 1952 Book of ASTM Standards, Part 4.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following two standards, as appended hereto,[3] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for:

Creosote (D 390 – 49), and
Creosote-Coal Tar Solution (D 391 – 50).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives[2] be continued in their present status without revision pending further study:

Tentative Methods of Test for:

Ash in Wood (D 1102 – 50 T),
Alpha Cellulose in Cellulosic Materials (D 1103 – 50 T),
Holocellulose in Wood (D 1104 – 50 T),
Preparation of Extractive-free Wood (D 1105 – 50 T),
Lignin in Wood (D 1106 – 50 T),
Alcohol-benzene Solubility of Wood (D 1107 – 50 T),
Ether Solubility of Wood (D 1108 – 50 T),
One Per cent Caustic Soda Solubility of Wood (D 1109 – 50 T),
Water Solubility of Wood (D 1110 – 50 T), and
Methoxyl Groups in Wood and Related Materials (D 1165 – 51 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (Lyman W. Wood, chairman) has given further thought and discussion to the Tentative Specification for Round Timber Piles (**D 25 – 52 T**), recommending it for continuance as a tentative revised in accordance with agreements reached. The committee is giving further study to the subject of working stresses and stress grades of lumber in connection with the contemplated revision of Tentative Methods for Establishing Structural Grades of Lumber (**D 245 – 49 T**).

Subcommittee II on Laminated Timber (F. J. Hanrahan, chairman) has encountered no developments significant enough to justify revision or changes in the Tentative Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (**D 1101 – 50 T**). It is recommended for adoption as standard as noted earlier in this report.

Subcommittee VI on Timber Preservatives (R. H. Bescher, chairman).—Two existing standard specifications, Creosote (**D 390 – 49**), and Creosote-Coal Tar Solution (**D 391 – 50**), have been reviewed and with slight modifications are recommended for immediate adoption as standard without reversion to tentative. In addition the subcommittee has developed four new tentatives for Copperized Chromated Zinc Chloride and Pentachlorophenol. These are recommended for publication as noted earlier in this report. During the year a sectional group was established to study soluble material in creosote other than benzol soluble material.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The proposal to study working stresses for wood poles, which will involve considerable testing of full-sized poles, has been given thought and study by the subcommittee. Costs and possible savings have been analyzed and four regional working groups have been selected to raise funds which will total

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[3] The revised specifications were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 4.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

$150,000. Reports are expected from the working groups by July 1, 1953.

Subcommittee VIII on Modified Wood and Wood-Base Material (W. G. Youngquist, chairman).—A tentative draft of a specification for compreg, a resin-impregnated compressed wood, was prepared and discussed. The draft will be revised in the light of the discussion and reviewed by subcommittee members.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—No revisions of present tentatives and standards are recommended. However, Committee D-7 recommends that Standard Methods for Testing Small Clear Specimens of Timber (**D 143 – 52**) be submitted to the American Standards Assn. for consideration as an American Standard.

Subcommittee XII on Fire-Retardant Wood (W. H. Fulweiler, chairman).—Any further progress of this subcommittee is largely contingent on developments in Committee E-5 on Fire Tests of Materials and Construction.

Subcommittee XV on Structural Fiberboards (Wayne C. Lewis, chairman) was organized during the year and its membership selected. The scope of the subcommittee was agreed upon and work will now get under way.

This report has been submitted to letter ballot of the committee, which consists of 81 voting members; 58 members returned their ballots, of whom 56 voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

L. J. MARKWARDT,
Chairman.

L. W. SMITH,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS*

Committee D-8 on Bituminous Waterproofing and Roofing Materials held two meetings during the year: one in New York, N. Y., June 25, 1952, the other in Detroit, Mich., March 4, 1953.

The committee suffered loss through the death of J. S. Miller. He had been a member of the Society since 1903 and a member of Committee D-8 since 1927. He served on a number of subcommittees and was chairman of Subcommittees III and X. From 1944 to 1950 Mr. Miller was chairman of Committee D-8.

The committee also suffered loss through the resignation of J. S. Crandell.

The membership consists of 75 voting members, composed of 46 producers, 12 consumers, and 17 general interest members.

All of the standards and tentatives for which Committee D-8 is responsible have been reviewed, and recommendations concerning them are set forth in this report.

REVISION OF TENTATIVE

The Committee recommends that the *Tentative Specifications for Asphalt Roofing Surfaced with Mineral Granules* (**D 249 - 50 T**)[1] be revised as indicated and continued as tentative:

Title.—Change to read "Asphalt *Roll* Roofing Surfaced with Mineral Granules."

Delete all reference to "32-in. roofing."

Section 6 (b), line 4.—Change "bare lapping edge" to "selvage."

Section 10 (a), lines 3, 4, and 5.—Change "bare lapping edge" to "selvage."

Table I.—Physical Requirements of Asphalt Roofing:

The change in weight basis from 108 to 100 sq ft makes necessary a number of changes, the new values being indicated below. Other items remain the same:

	Max, lb	Min, lb
Weight of any 100 sq ft of roofing in the shipment (granule surfaced portion)		74.0
Weight of dry felt per 100 sq ft		10.0
Weight of saturant (soluble in CS_2) per 100 sq ft[a]		16.0
Weight of weather side coating per 100 sq ft	32.4	14.8
Weight of mineral matter per 100 sq ft passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve		18.5

[a] The weight of saturant per 100 sq ft shall be not less than 1.6 times the weight of the dry felt.

TENTATIVE REVISION OF STANDARDS

The committee recommends the publication of tentative revisions of the following two standards:

Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (**D 224 - 52**):[1]

Title.—Change to read "Specifications for Asphalt *Roll* Roofing Surfaced with Powdered Talc or Mica."

Delete all reference to "32-in. roofing."

Section 6 (b).—Change to read "The coating shall be applied uniformly and in approximately equal thickness on both sides and up to the edges of the sheet. The weather side shall be surfaced with talc or mica. The reverse side shall be surfaced with the same or other suitable materials to prevent the roofing from sticking in the package."

Table I.—Physical Requirements of Asphalt Roofing:

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 3.

The change in weight basis from 108 to 100 sq ft makes necessary a number of changes, the new values being indicated below. Other items remain the same:

	65-lb Grade		55-lb Grade	
	Max	Min	Max	Min
Weight of any 100 sq ft of roofing in shipment, lb.....		55.6		46.3
Weight of dry felt per 100 sq ft, lb....................		12.1		10.0
Weight of saturant (soluble in CS_2) per 100 sq ft[a], lb.....		19.4		16.0
Weight of coating and surface mineral matter per 100 sq ft, lb......................		16.7		16.7

[a] The weight of saturant per 100 sq ft shall be not less than 1.6 times the weight of the dry felt.

Specifications for Wide Selvage Asphalt Roofing Surfaced with Mineral Granules **(D 371 – 51)**:[1]

Title.—Change to read "Specifications for Wide Selvage Asphalt *Roll* Roofing Surfaced with Mineral Granules."

Delete all reference to "32-in. roofing."

Table I.—Physical Requirements for Asphalt Cap Sheet:

The change in weight basis from 108 to 100 sq ft make necessary a number of changes, the new values being indicated below. Other items remain the same.

	45-lb Grade		55-lb Grade	
	Max	Min	Max	Min
Weight of any 100 sq ft of roofing in shipment, lb.........		37.0		46.3
Weight of dry felt per 100 sq ft, lb......................		6.6		10.0
Weight of saturant (soluble in CS_2) per 100 sq ft[b], lb......		9.3		16.0
Weight of weather side coating per 100 sq ft[a], lb	32.4	14.8	32.4	14.8
Weight of weather side mineral matter passing No. 6 (3360-micron) sieve and retained on No. 100 (149-micron) sieve per 100 sq ft[a], lb.....		18.5		18.5

[b] The weight of saturant per 100 sq ft shall be not less than 1.4 times the weight of the dry felt for the 45-lb grade, and 1.6 times the weight of the dry felt for the 55-lb grade.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following tentatives, which have been published by the Society for two or more years, be continued without revision since they are being reviewed for possible revisions to be recommended before the next Annual Meeting:

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, Shingles (D 228 – 48 T)

Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Material (D 529 – 39 T)

Tentative Methods of Testing Asphalt Base Emulsions for Use as Protective Coatings for Built-Up Roofs (D 1167 – 51 T).

EDITORIAL CHANGES

The committee recommends editorial changes in the following standards:

Specifications for Asphalt Shingles Surfaced with Mineral Granules **(D 225 – 51)**:[1]

Table I.—Physical Requirements of Asphalt Shingles:

The change in weight basis from 108 to 100 sq ft makes necessary a number of changes, the new values being indicated below. Other items remain the same.

	Type I		Type II		Type III	
	Max	Min	Max	Min	Max	Min
Weight of roofing per 100 sq ft (individual package), lb.........		76.9		87.0		80.1
Weight of exposed area per 100 sq ft, lb.....		76.9		87.0		100.0
Weight of dry felt per 100 sq ft, lb.........		10.0		12.5		10.0
Weight of saturant per 100 sq ft, lb		17.5		21.9		17.5
Weight of weather side coating per 100 sq ft, lb................	32.4	14.8	42.6	14.8	55.6	18.5
Weight of mineral matter per 100 sq ft passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve, lb		18.5		18.5		18.5

[b] The weight of saturant per 100 sq ft shall be not less than 1.75 times the weight of the dry felt.

Specification for Asphalt for Use in Constructing Built-Up Roof Covering **(D 312 – 44)**:[1]

Table I.—On the last item reading "Coarse particles retained on No. 200 (74-micron) sieve as percentage of matter insoluble in carbon disulphide, per cent. —Max 12," the following footnote should be added: "This limit applies only on mineral stabilized or native asphalt." In lines 16 and 21, the word "Filled"

shall be changed to read "Mineral Stabilized," and in lines 17 and 22 "Unfilled asphalt" shall be changed to read "Asphalt without Mineral Stabilizer."

Standard Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (**D 146 – 47**):[1]

Section 12 (a), lines 16, 17 and 18.—Change from "in which the clamps are attached to swivels free to move in any direction," to read "the clamps shall have sufficient freedom of movement to permit alignment in one plane."

Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 226 – 47**):[1]

Section 9.—Change "Saturant in moisture-free felt, min, per cent by weight" to read "Weight of Saturant per 100 sq ft for the 15-lb type 7.3 lb min and for the 30-lb type 15.00 lb min", with a footnote following the table reading "Weight of Saturant per 100 sq ft shall be not less than 1.40 times the weight of the dry felt for the 15-lb type and 1.50 times the weight of the dry felt for the 30-lb type."

Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (**D 227 – 47**):[1]

Section 8.—Change "Saturant in moisture-free felt" to read "Weight of saturant per 100 sq ft 7.3 lb, min" with a footnote following the table reading "Weight of saturant per 100 sq ft shall not be less than 1.40 times the weight of the dry felt."

Standard Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (**D 250 – 47**):[1]

Section 9.—Change "Saturant in moisture-free felt, min, per cent by weight" to read "Weight of saturant per 100 sq ft for 15-lb type is 3.6 lb min, and for 30-lb type it is 8.8 lb min" with a footnote following the table reading "Weight of saturant per 100 sq ft shall not be less than 0.40 times the weight of dry felt for the 15-lb type and 0.50 times the weight of the dry felt for the 30-lb type."

Standard Specifications for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (**D 655 – 47**):[1]

Section 9.—Change "Saturant in moisture-free felt, min, per cent by weight" to read "Weight of saturant per 100 sq ft for 20-lb type is 3.40 lb min and for 50-lb type is 8.8 lb min," with a footnote following the table reading "Weight of saturant per 100 sq ft shall not be less than 0.4 times the weight of dry felt for the 20-lb type and 0.5 times the weight of the dry felt for a 50-lb type."

A Proposed Method of Test for Contact Compatibility of Bituminous Materials is recommended for publication as information and comments, as appended hereto.[2] Data secured in a round-robin test utilizing the procedure are given in the Appendix in support of the method.[3]

The recommendations appearing in this report have been submitted to letter ballot of the committee; the results will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 66 members; 59 members have returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

H. R. SNOKE,
Chairman.

G. W. ROBBINS,
Secretary.

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[2] See p. 411.
[3] See p. 410
[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

RESULTS OF TESTS FOR INCOMPATIBILITIES OF BITUMINOUS MATERIALS

A cooperative test series employing the Proposed Method of Test for Contact Compatibility of Bituminous Materials, appended hereto,[1] has been carried out among the members of Subcommittee XIV, utilizing six saturants and three coatings. The results of these tests, which are given in Table I, show that incompatibilities of less than 0.2 mm can be checked to ±0.1 and from there to 0.5 mm with ±0.2 variation. Since it is normally desired that asphalts for the roofing industry be substantially wholly free from any incompatibility, the wider variations at high values are seemingly irrelevant.

Comments from any individual using this method will be appreciated.

TABLE I.—RESULTS OF TESTS FOR INCOMPATABILITY OF BITUMINOUS MATERIALS.
All Tests at 50 C (122 F) for 72 Hr.

Saturant	Lab. A	Lab. B	Lab. C	Lab. D	Lab. E	Lab. F	Lab. G	Lab. H	Lab. I	Lab. J	Min.	Max.	Average
Coating C													
31	0.1	0.0	Tr.	0.1	0.1	0.2	0.0	0.2	0.2	0.0	0.0	0.2	0.09
32	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00
33	0.7	0.4	0.3	0.5	0.3	0.6	0.7	0.4	0.4	0.1	0.1	0.7	0.45
34	0.2	0.1	0.3	0.3	0.2	0.2	0.3	0.4	0.3	0.2	0.1	0.4	0.25
35	0.7	0.1	0.3	0.5	0.2	0.5	0.0	0.4	0.2	0.0	0.1	0.7	0.29
36	0.1	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.02
Coating D													
31	0.0	0.0	Tr.	0.1	Tr.	0.1	0.0	0.1	0.1	0.0	0.0	0.1	0.04
32	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00
33	0.5	0.0	0.3	0.2	0.2	0.4	0.3	0.3	0.4	0.5	0.0	0.5	0.31
34	0.2	0.0	0.1	0.0	0.1	0.2	0.2	0.2	0.2	0.0	0.0	0.2	0.12
35	0.1	0.0	0.2	0.2	0.1	0.2	0.2	0.2	0.2	0.0	0.0	0.2	0.14
36	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.01
Coating E													
31	1.0	0.6	0.7	0.7	0.7	0.8	1.0	0.7	0.7	0.4	0.6	1.0	0.73
32	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00
33	1.2	0.8	1.2	1.0	1.0	1.0	1.0	0.9	1.0	1.0	0.8	1.2	1.01
34	1.0	0.5	0.9	0.6	0.8	0.6	1.0	0.8	0.7	0.5	0.5	1.0	0.74
35	0.7	0.7	0.8	0.6	1.0	1.0	0.9	0.6	0.8	0.5	0.6	1.0	0.76
36	0.2	0.3	0.2	0.0	0.1	0.1	0.4	0.2	0.3	0.1	0.0	0.3	0.19

AVERAGES OF INCOMPATIBILITIES REARRANGED IN ASCENDING ORDER.

Coating	Saturation	Average	Min.	Max.	Standard Deviation
D	32	0.00	0.0	0.0	0.00
C..........	32	0.00	0.0	0.0	0.00
E..........	32	0.00	0.0	0.0	0.00
D	36	0.01	0.0	0.1	0.03
C..........	36	0.02	0.0	0.1	0.04
D	31	0.04	0.0	0.1	0.05
C..........	31	0.09	0.0	0.2	0.09
D	34	0.12	0.0	0.2	0.09
D	35	0.14	0.0	0.2	0.08
E..........	36	0.19	0.0	0.4	0.11
C..........	34	0.25	0.0	0.3	0.08
C..........	35	0.29	0.0	0.7	0.22
D	33	0.31	0.0	0.5	0.14
C..........	33	0.45	0.1	0.7	0.17
E	31	0.73	0.4	1.0	0.17
E..........	34	0.74	0.5	1.0	0.18
E..........	35	0.76	0.5	1.0	0.16
E..........	33	1.01	0.8	1.2	0.11

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[1] See p. 411.

PROPOSED METHOD OF TEST FOR CONTACT COMPATIBILITY OF BITUMINOUS MATERIALS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test provides a means for evaluating contact compatibility between bituminous materials. It is generally used to determine compatibility between the saturant and coating used in the manufacture of prepared roofings. In the following description of the method, coating and saturant will be referred to, but it is understood that comparable bituminous materials may be substituted where this test procedure seems applicable.

Outline of Method

2. A small drop of molten saturant is placed on the freshly talced surface of the coating, and compatibility is judged by the degree to which an oily ring develops in the talc surrounding the drop.

Apparatus

3. (*a*) *Container*, lid of 3-oz ointment box, or equivalent container.

(*b*) *Analytical Balance* having an accuracy of ±1.0 mg.

(*c*) *Sieve*, No. 325 (44 micron).[3]

(*d*) *Dropping Device*, a fine wire, 1.0 mm (0.04 in.) in diameter.

(*e*) *Oven*, constant-temperature, capable of maintaining the test temperature within ±1.0 C (±1.8 F).

(*f*) *Small Scale*, graduated in 0.5-mm divisions.

(*g*) *Magnifying Glass*, approximately 4×.

(*h*) *Talc*, pure, of the soapstone variety, ground so that at least 70 per cent passes the No. 325 sieve,[3] and oven-dried at 110 C.

NOTE.—When this procedure is being used in purchase specifications, both the purchaser and the seller shall use the same talc.

Procedure

4. (*a*) Melt a portion of the coating at the minimum temperature required to render it fluid, stirring with the fine wire to eliminate bubbles, and pour it into the clean 3-oz tin lid to a thickness of 3.0 to 6.0 mm. Pour carefully, so as to provide a substantially smooth surface free of bubbles and other surface blemishes. After the coating material has cooled, determine the weight and surface area of the filled container.

(*b*) Make a preliminary dusting of talc by covering the coating surface with talc and removing the excess, nonadherent dust by inverting the container and dropping it approximately 2.5 cm onto the table top.

(*c*) Make a final application of talc through the No. 325 sieve, held 2.0 to 2.5 cm above the surface of the coating, by gently shaking or tapping the sieve until a smooth, uniform film of talc, weighing 0.004 g ±10 per cent per square centimeter, has been applied.

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[1] This proposed method is under the jurisdiction of ASTM Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Published as information, June, 1953.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (ASTM Designation: E 11), 1952 Book of ASTM Standards, Part 3.

(*d*) Place three drops of molten saturant, each about 3.0 ±0.5 mm in diameter, on the talced surface of the coating. Form the drops by plunging the wire into the molten saturant and, after the excess has drained off, allowing suitably sized drops to fall on the talc from a sufficient height to form substantially spherical drops.

(*e*) Place the prepared specimen in the test oven, so regulated as to maintain the specimen at the recommended test temperature, within ±1.0 C (±1.8 F), for 72 hr (Note). After cooling, examine the specimen and measure any oily ring formed around the periphery of the drop, recording its width to the nearest 0.1 mm. Measure the width from the line of contact of talc and drop to the outer limit of the oil ring.

NOTE.—The recommended test temperature for roofing materials is 50 C (122 F), but this may be altered to accommodate variations in the bitumens being tested.

Report

5. Report the results of the compatibility test as the width in millimeters of the ring developed at the specified test temperature.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in New York City, June, 1952, held in connection with the Annual Meeting of the Society; in Boston, Mass., on October 29 to 31, 1952; and at Pocono Manor, Pa., on March 24 to 27, 1953.

At these meetings action was taken on several new methods, new recommended practices, revision of existing tentatives, and changes in some of the standards under the jurisdiction of Committee D-9. Also, progress was made on other projects under study by the various subcommittees, as mentioned later in the report.

At its meeting on October 30, 1952, Committee D-9 unanimously elected W. A. Evans as Honorary Member of the committee in appreciation of his long-term service and many contributions to the committee's work.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-9 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Methods of:

Testing Varnishes Used for Electrical Insulation (D 115 - 52a T),

Testing Non-Rigid Polyvinyl Tubing (D 876 - 51 T),

Revision of Tentative Specifications for:

Phenolic Molding Compound (D 700 - 49 T), (Submitted Jointly with Committee D-20 on Plastics), and

Non-Rigid Polyvinyl Tubing (D 922 - 47 T).

These recommendations were accepted by the Administrative Committee on Standards on December 12, 1952, and the revised tentatives appear in the 1952 Book of ASTM Standards, Part 6.

NEW TENTATIVE

Committee D-9 recommends that the proposed Tentative Method of Test for Corrosive Sulfur in Electrical Insulating Oils, as appended hereto,[1] be accepted for publication as tentative. This method covers a procedure for determining sulfur and corrosive sulfur in oils used in transformers, switch gears, circuit breakers, and other electrical insulation.

REVISION OF TENTATIVES

Tentative Methods of Testing Pressure-Sensitive Adhesive Tapes Used for Electrical Insulation (**D 1000 - 48 T**).[2]—The committee recommends the following revision of these methods:

Section 1 (*a*).—Insert the word "coated" between the words "adhesive" and "tapes."

Section 4 (*b*).—Change the conditioning temperature from "77 F" to "73.4 F (23 C)" to conform to the temperature of standard laboratory atmosphere.

Section 19.—In Paragraph (*a*), revise the second sentence to read "The machine shall have one stationary head and one movable head and shall be equipped with a tension weighing device graduated and accurate to 0.1 lb or less (preferable 0.05 lb or less) per scale division."

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*Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 6.

[2] 1952 Book of ASTM Standards, Part 6.

Revise Paragraph (*b*) to read as follows:

(*b*) *Steel Strip.*—Cold-rolled stainless steel strip type 302 shall be used as prescribed in the Standard Specifications for Corrosive-Resisting Chromium-Nickel Steel, Plate, Sheet, and Strip (ASTM Designation: A 167). The stainless steel strip shall be $\frac{1}{16}$ in. thick by 2 in. wide by a minimum length of 5 in., finished with a No. 150 grit belt as standardized by the stainless steel industry.[a]

In Paragraph (*c*) replace Fig. 1, which shows the roller for the adhesion strength test, by the accompanying Fig. 1, which is a new design that eliminates the effect of pressure on the handle.

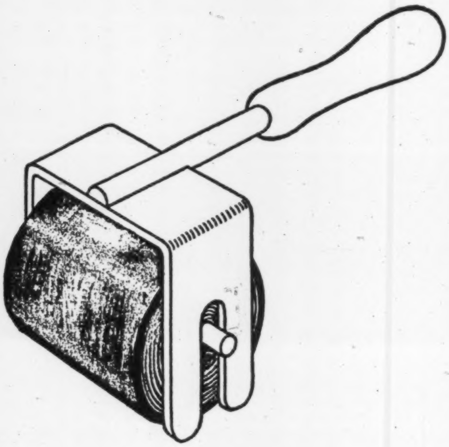

FIG. 1.—Suggested Construction of Roller for Adhesion Strength Test.

Revise the second sentence to read "The weight of the roller proper which applies pressure to the specimen shall be 4.5 ± 0.1 lb. It shall be constructed so that the weight of the handle is not added to the weight of the roller during use."

Section 26.—Delete Footnote 6 and the reference to this footnote in the heading preceding this section.

Section 28 (*c*).—Change the conditioning temperature from "25 ± 1 C" to "23 ± 1.1 C" to conform to the temperature of the standard laboratory atmosphere, and add "relative" before "humidity."

Add another paragraph to the Note at the end of the section, as follows: "It is advisable to lag the test chamber with thermal insulation in order to minimize internal temperature fluctuations caused by temperature changes and/or drafts in the surrounding air."

Appendix.—Delete Appendix I on "Tensile Strength Method for Determining Electrolytic Corrosion."

Tentative Specifications for Natural Muscovite Mica Based on Visual Quality (**D 351 - 52 T**).[2]—The committee recommends the following revisions of these specifications:

Section 1.—Master reference standards for waviness for use in the visual grading of mica have now been selected. It is accordingly recommended that Note 3 in Section 1 be revised to read as follows:

NOTE 3.—In order to clarify the verbal descriptions of visual quality of natural muscovite mica, a set of Master Standards of Waviness,[a] representing the full range of wave variations for visual quality levels of Good Stained and better has been selected.

Section 4.—Make the following changes in the terminology and definitions:

Delete the term "Processed Mica" and substitute in its place "Unmanufactured Mica."

Delete the term "Strip" and the entire sentence that follows.

Delete the term "Hairlines" and in its place substitute "Hairline Cracks." Also delete the word "Surface" in the sentence following this term.

In Note 3 under the term "Inclusion," delete the word "reflected" and in its place substitute the word "transmitted."

In Note 2 under the term "Stains," delete the word "reflected" and substitute the word

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[a] A steel strip that has been found satisfactory for this purpose can be purchased from the United States Steel Corp.

[a] At the present time, the original set of Master Standards of Waviness is the only one available and can be inspected at ASTM Headquarters, 1916 Race St., Philapelphia 3, Pa.

"transmitted."

Add a new term "Crystallographic Discoloration" which shall follow the term "Stains." Add the following definition: "Crystallographic Discoloration appears as bands of lighter or darker shades of the basic color of the block of mica. Such bands generally are parallel to the crystallographic faces of the crystal from which the block was separated."

Section 5(a).—Delete all of Paragraph (*a*) and substitute the following:

For full trimmed mica—all mica blocks, thins and condenser films shall be fully trimmed to remove all cracks, holes, reeves and crossgrains according to the quality desired. Trimming should follow the natural contour of the mica. As far as possible, all marginal cracks should be removed by recutting. The total area of each piece for grade 4 and larger shall be not more than 1.54 times the area of the largest usable rectangle or rectangular yield of 65 per cent. For grades 5 and smaller the total area of each piece shall be not more than two times the area of the largest usable rectangle or rectangular yield of 50 per cent. The usable area is the area of maximum rectangle obtainable. If limitation as to the size, number and frequency of "V" cuts is desired, it shall be subject to agreement between the purchaser and the vendor.

Table I.—Change the dimension of one side for Grade A-1 (Special) from 4 to 3½.

Section 9(a).—Add a new symbol *h* under Table II and have it appear at the head of column 1 next to the heading "Clear Uniform Basic Color[h]."

Add the following description after *h* under the table:

[h] Crystallographic discoloration is permitted to a limited extent in V-4 Good Stained quality. It is not permitted in qualities higher than V-4 Good Stained. In qualities below V-4 Good Stained it is permitted without limit.

Add a new visual quality designation to the Table as follows:

ASTM Visual Quality	Commercial Designation
V-11	Densely Stained

Table II.—Add an additional classification V-11, Densely Stained and provide check marks under all columns except columns headed Black Stains (Mineral)[g] and Red Stains (Mineral)[e] under which place x marks.

Appendix I.—Add the following Verbal Description of Visual Quality:

V-11. Densely Stained.—Soft and may contain all heavy stains and inclusions and other defects, heavy waves, cracks and buckles.

Appendix III.—Revise Table ME1-4.01—Classification of Muscovite Splittings—to bring it in line with revision of Publication No. ME1—1952, Standards for Manufactured Electrical Mica, National Electrical Manufacturers Association.

Tentative Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation (**D 202 - 52 T**).[2]—The following four statements on significance of tests for tensile strength, tearing strength, ash, and dielectric strength are recommended for inclusion in these methods, the statement to appear at the beginning of the respective test procedure, with the present section numbers changed accordingly:

TENSILE STRENGTH

12. *Definition*.—Tensile strength of paper used for electrical insulation is defined as the force required to rupture a test specimen of definite width and length, cut from either or each principal direction of the paper, conditioned and tested as prescribed.

Tensile strength is customarily expressed as the force per unit width (for example, kg per 1.5 cm or lb per 1 in.)

If the thickness of the test specimen is measured, the Tensile Strength, as force per unit area, can be compared.

13. (*a*) *Significance*.—The results of the test are suitable:

(*1*) For acceptance; product control; research; or referee testing, and

(*2*) For measuring serviceability (suitability) of papers which in use are subjected to direct tensile stresses such as longitudinal pull on tapes, sheets, etc.

(*b*) Tensile strength of paper is influenced by:

(*1*) The kind, quality, and treatment of the paper fibers,
(*2*) The formation of the sheet on the paper making machine, and
(*3*) The treatment of the sheet after formation (machine wet pressing and calendering).

TEARING STRENGTH

16. *Definition.*—Tearing strength of paper used for electrical insulation is defined as the grams of force required to tear one thickness of paper as prescribed.

17. (*a*) *Significance.*—The results of the test are suitable:

(*1*) For acceptance; product control; research; or referee testing, and
(*2*) For measuring serviceability (suitability) of papers which in use are subjected to tear strain.

(*b*) Tearing strength of paper is influenced by:

(*1*) The kind, quality, and treatment of the paper fibers,
(*2*) The formation of the sheet on the paper making machine, and
(*3*) The treatment of the sheet after formation (coating, saturating, etc.)—(machine wet pressing and calendering).

ASH

36. *Definition.*—Ash is the solid residue remaining after complete combustion of the paper under specified conditions. It is expressed as a percentage of the weight of the original specimen, dried as specified.

37. *Significance.*—The ash determination is a relatively simple and convenient way of detecting the presence of mineral filling, coating, or pigmenting materials in paper. Such materials are normally to be avoided in papers for electrical insulation. The procedure is suitable for control testing, research, and referee work.

DIELECTRIC STRENGTH

103. *Definition.*—The dielectric strength of untreated paper is the voltage gradient, at which breakdown occurs when the paper is tested under the prescribed conditions. It is usually expressed in volts per mil and is obtained by dividing the puncture voltage by the thickness of the paper specimen tested.

104. *Significance.*—For paper to be used in the untreated state, this test gives some indication of the electrical service strength. For the paper to be subsequently treated, this test has value as a quality control test. It is also suitable for use as a specification test.

The committee recommends that the Bursting Strength Test be revised as follows, which provides for use of ASTM Method D 774 as Procedure A for papers bursting at not over 200 psi hydrostatic pressure and to designate the present test as revised as Procedure B for papers bursting at more than 200 psi. The revised bursting strength procedures are as follows:

BURSTING STRENGTH

19. *Definition.*—Same as present Section 15.

20. *Significance.*—Same as present Section 16.

Procedure A for Papers Bursting at Not Over 200 psi Hydrostatic Pressure

21. *Procedure.*—The bursting strength shall be determined in accordance with Standard Method of Test for Bursting Strength of Paper (ASTM Designation: D 774), except that the material shall be sampled in accordance with Section 2 of these Methods D 202.

Procedure B for Papers Bursting at More Than 200 psi Hydrostatic Pressure

22. *Apparatus.*—The testing machine shall conform to the following requirements:

(*a*) *Clamps.*—These shall be similar in all respects to those used on the machine specified in the Standard Method of Test for Bursting Strength of Paper (ASTM Designation: D 774).

(*b*) *Rubber Diaphram.*—This shall be pure gum rubber, free from mineral loading material, and shall have the special shape and thickness measurements shown in Fig. 1 (the accompanying Fig. 2).

(*c*) *Motor.*—The hydrostatic pressure applied to the underside of the rubber diaphragm until the test specimen bursts shall be generated by a motor-driven piston forcing a liquid (usually glycerine or hydraulic brake fluid) into the pressure chamber of the apparatus at the rate of 170 ± 0.2 ml (about 10.4 cu in.) per min.

(*d*) *Pressure Reading Gages.*—The maximum pressure reading gages with dials, preferably 5 or more in. in diameter, shall indicate the bursting pressures in pounds per square inch with the folowing accuracy:

Pressure, psi	Accuracy, psi
0 to 300	3.0
0 to 600	6.0
0 to 1000	10.0
Above 1000	20.0

NOTE.—The gages should be calibrated in a dead-weight tester. Where the gages are used more than once weekly, they should be calibrated weekly. When used weekly or less often, the gages should have been calibrated within 30 days of their use.

23. *Test Specimen.*—Same as present Section 18.

24. *Procedure.*—Same as present Section 19.

25. *Report.*—Same as present Section 20.

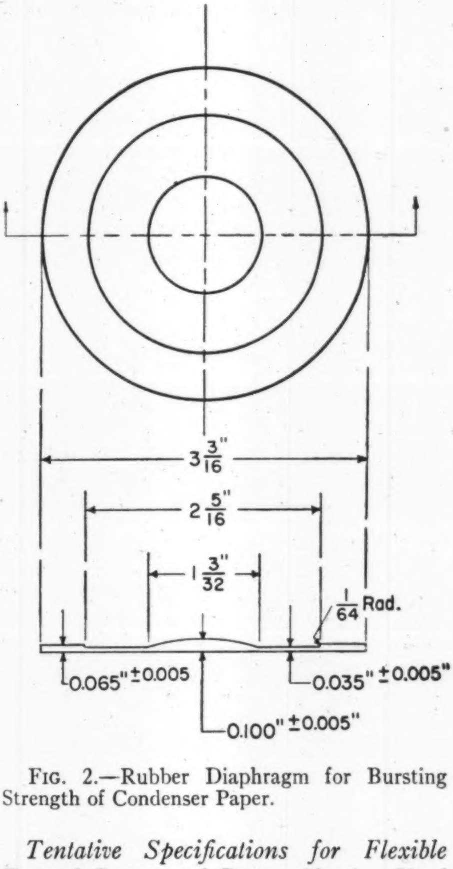

FIG. 2.—Rubber Diaphragm for Bursting Strength of Condenser Paper.

Tentative Specifications for Flexible Treated Cotton and Rayon Sleeving Used in Electrical Insulation (**D 372 - 47 T**).[2] It is recommended that these specifications be revised by changing the descriptions of the three classes of material A, B, and H, and the inclusion of a new Table I on identification of grades and summary of performance characteristics. The revised specifications are no longer restricted to use of only cotton and rayon; also the color requirements have been revised. The specifications revised are appended hereto.[3]

Tentative Methods of Testing Electrical Insulating Oil (**D 117 - 50 T**).[2] The committee recommends the addition of a footnote under Section 16, in reference to the procedure for determining interfacial tension, to read as follows:

> If desired for technical reasons or to expedite routine testing, preliminary filtering of the sample may be omitted. The results obtained on unfiltered samples shall be reported as interfacial tension (unfiltered).

Tentative Methods of Testing Askarels (**D 901 - 52 T**).[2]—The committee recommends the inclusion in these methods of the statements appended hereto[3] on "definition" and "significance" of the following test procedures: Sampling, Specific Gravity, Viscosity, Fire Point, Arc-formed Gases, Refractive Index, Power Factor and Dielectric Constant, and Dielectric Strength.

TENTATIVE REVISION OF STANDARD

The committee recommends for publication the following tentative revision of the Standards Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (**D 350 - 48**).[2]

Section 1.—Change "varnished tubing and saturated sleeving" to "treated sleeving."

Note.—Revise the tentative revision[4] under Section 1, issued December, 1944, to read as follows:

> **NOTE.—The term "flexible treated sleeving" as used in these methods refers to a flexible tubular product made from braided cotton, rayon, nylon, glass, or other fibers and impregnated, or impregnated and coated with a film of varnish, lacquer, or other electrical insulating materials.**

Section 3.—Change the first sentence to read: "The test specimens for dielectric strength tests shall be conditioned for 96 hr in air maintained at a temperature of 23 ± 1.1 C and at a relative humidity of 96 ± 2 per cent."

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[3] The revised specifications were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 6.

[4] 1952 Book of ASTM Standards, Part 6, p. 1082.

Section 14 (*a*).—Revise the aging test procedure to read as follows:

14. (*a*) The specimens shall be placed in a uniformly heated analytical-type electric oven in which the temperature is maintained at:

Class AA-1, AA-2, AB-1, AB-2 BA-1, BA-2, BB-1, BB-2	$105 {}^{+5}_{-0}$ C $221 {}^{+9}_{-0}$ F
Class HA-1, HB-1	20 ± 5C 392 ± 9F

Two specimens shall be removed at the end of 24 hr and every 24 hr thereafter until failure occurs. After cooling to room temperature, each specimen shall slowly be bent 180 deg over a rod having a diameter in accordance with the following:

Size of Tubing or Sleeving	Diameter of Test Rod. in.
1 to $\frac{3}{4}$ in., incl.	$\frac{7}{16}$
$\frac{5}{8}$ to $\frac{3}{8}$ in., incl.	$\frac{3}{8}$
Nos. 0 to 5, incl.	$\frac{5}{16}$
No. 6 and smaller	$\frac{1}{4}$

New Test.—Add the following new test procedures for "Resistance to Transformer Oil" to appear as Sections 23 to 25:

23. *Test Specimen.*—Three specimens, 3 in. (7.6 cm) in length, shall be cut from the samples obtained in accordance with Section 2.

24. *Procedure.*—The specimens shall be immersed for 48 hr in transformer oil maintained at a temperature of 105 C. At the end of this time, the specimens shall be removed from the oil, and examined after removal of excessive oil.

25. *Examination.*—The film shall be examined for disintegration or swelling.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentative methods be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (**D 902 - 52 T**)[2] with the following changes which emphasize reference to similar provision in Methods D 149 for Dielectric Strength:

Change Section 28 to read:

28. The report shall include the following:

(*1*) The test method used for determining the dielectric strength,

(*2*) The average, maximum and minimum dielectric breakdowns in volts for each test method,

(*3*) The average, maximum and minimum thicknesses of the sample as determined in Section 11, and

(*4*) The average dielectric strength in volts per mil obtained by dividing the average dielectric breakdown in (*2*) by the average thickness in (*3*).

Section 31.—Add the following note at the end of this section:

NOTE.—Guarded foil electrodes as described in Section 13 of the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (ASTM Designation: D 150) may be used for measurements at room temperatures.

Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (**D 618 - 51 T**).[2]—These methods are submitted jointly with Committee D-20 on Plastics and are recommended for adoption as standard with the addition of the following sentence to Note 2 in Section 4: "Shorter conditioning times may be used for their specimens provided equilibrium is substantially obtained."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuance of the following tentatives without change for an additional year, while work is in progress:

Tentative Specifications for:

Black Bias-Cut Varnished Cloth and Varnished Cloth Tape Used for Electrical Insulation, (D 373 - 51 T),
Non-Rigid Vinyl Chloride Plastics (D 744 - 49 T),

Tentative Methods of Test for:

Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 - 48 T),
Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 47 T),
Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 47 T),
High-Voltage, Low-Current Arc-Resistance of Solid Electrical Insulating Materials (D 495 - 48 T),
Heat Distortion Temperature of Plastics (D 648 - 45 T),
Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials (D 669 - 42 T),
Sludge Formed in Mineral Transformer Oil (D 670 - 42 T),
Glass-Bonded Mica Used as Electrical Insulation (D 1039 - 50 T),
Hydrocarbon Waxes Used for Electrical Insulation (D 1168 - 51 T), and

Tentative Practice for:

Purchase of Uninhibited Mineral Oil for Use in Transformers and in Oil Circuit Breakers (D 1040 - 49 T).

CONTINUATION OF TENTATIVE REVISION

The committee recommends that the tentative revision of the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (**D 149 - 44**) be retained for an additional year, while work is in progress.

EDITORIAL CHANGE

The committee recommends that the Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (**D 619 - 52 T**) and the Specifications for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (**D 710 - 52 T**) be editorially revised by adding the phrase "in varying degrees, depending upon the grade of fibre" at the end of the definition of vulcanized fibre.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (A. H. Haroldson, chairman).—Sections A and B of this subcommittee cooperated in rewriting the Tentative Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 52a T). Sections A and B also cooperated in writing a definition for electrical insulating varnishes, which was submitted to members of the subcommittee for comment. The comments received will be referred to Section K for further study.

Section C members have developed a simple and inexpensive constant temperature wax bath. It has been planned to conduct round-robin tests on set time of laminating varnishes using the new bath.

Section D has tentatively approved methods for the determination of specific gravity, viscosity, flash point, draining test, nonvolatile matter by weight, and dielectric strength of silicone varnishes. The methods will be rewritten and submitted to Subcommittee I for approval. Round-robin tests will be conducted on heat stability and drying time of silicone varnishes. Heat stability tests will be conducted on varnishes covering a range of properties in order to determine whether the proposed method can be used to separate different varnishes. A new method for the determination of drying time has been developed and will be studied in the round-robin testing program.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section G has cooperated with other sections to rewrite a test method for heat flexibility and to develop a method for determination of heat stability of silicone varnishes.

Section H has cooperated with other sections to rewrite the test for oil resistance and will cooperate in developing a method for determining the oil and chemical resistance of silicone varnishes.

Section J has proposed a method that will more accurately evaluate the heat stability of electrical insulating varnishes than the present heat flexibility test in Methods D 115. The method is based on the determination of the minimum breakdown voltage caused by film rupture due to elongation of the film.

Subcommittee III on Plates, Rods, Tubes, and Molded Materials (G. H. Mains, chairman).—In cooperation with Committee D-20 on Plastics, the Specifications for Thermosetting Molding Compounds (D 700 - 52 T) were revised last year to bring definitions and property values for the various grades up to date. The Methods of Test and Specification for Non-Rigid Polyvinyl Tubing (D 876 - 52 T and D 922 - 52 T) were also revised last year to add methods and property values on dielectric strength under humid conditions and on stress relief. An editorial revision of the definition of vulcanized fibre in Method D 619 and Specifications D 710 has been recommended by Subcommittee III to Committee D-9 to conform with action of NEMA and ASA. The subcommittee has cooperated with the Electrical Section of the Federal Specifications Board in revising the former Federal Specification HH-256. The new Federal Specification, LL-31, on Thermosetting Laminated Sheets, Tubes, and Rods coincides closely with ASTM Specification D 709 - 52 T.

The subcommittee is conducting letter ballots on the addition of rubber-modified phenolics to Specifications D 700, and on the clarification of the note on ammonia content, also on the adoption as standard of the Tentative Method of Test for Heat Distortion Temperature of Plastics (D 648 - 45 T), on a proposed procedure for cleaning samples of molded and laminated plastics prior to insulation tests, and on the desirability of adding a new section to the subcommittee to study the electrical properties of polyester molded and laminated products.

Current activities of the various sections include the following: Section G is studying the possibility of combining Methods D 551 and D 955 on measurement of mold shrinkage. Section J is reviewing the method of test and property values for dielectric strength of plastic tubing under humid conditions, to answer some criticism of the method; also round-robin tests on heat aging at 130 C, with a view to establishing a specification for high-temperature plastic tubing. Section Q is studying the revision of Methods D 229, D 348, and D 349 to conform with the lastest revisions of Methods D 638 and D 695 on tensile and compressive strength of plastics.

Section R is conducting round-robin tests on dissipation factor parallel with the laminations; also studies are being made on improving dielectric strength test methods for step-by-step dielectric strength tests on laminates. Section V is cooperating with Committee D-11 on Rubber Products on resolving discrepancies in the Specification for Gaskets (D 1170 - 51 T) and the Methods and Specifications for Vulcanized Fibre (D 619 - 52 T and D 710 - 52 T). Also studies are being conducted on a new method for zinc chloride in vulcanized fibre. Section W is continuing active work on significance statements for Methods D 348 and D 349.

Subcommittee IV on Liquid Insulation (F. M. Clark, chairman).—The activities of this subcommittee have included a wide variety of investigations pertaining to liquid dielectric materials with especial reference to mineral oil. Prominent among these studies are those relating to sludge formation in mineral oil and its evaluation in the base oil and in the inhibited oil, the detection and test for corrosive free sulfur and corrosive combined sulfur, the establishment of a testing procedure suitable for determining the amount of dissolved or suspended water in dielectric liquids, and the determination of the dielectric strength of viscous oils of petroleum origin.

The study of sludging phenomena in uninhibited mineral transformer oils has been continued, using the two test procedures described in Tentative Methods D 670 - 42 T, the oils tested being those taken from the transformers which have been in operation for as long as 10 or more years, as described in previous symposia publications on this subject. A critical survey of the efficiency of the two methods of test (sludge accumulation and high-pressure oxidation) is in progress.

Initial studies are in progress which have the objective of establishing a method suitable for the evaluation of the sludging characteristics of inhibited transformer oils.

Subcommittee IV has been represented in the activities of Technical Committee No. 10 of the International Electrotechnical Commission. It is expected that Committee No. 10 will submit to Subcommittee IV within the next year a method for consideration as an international testing standard for determining the sludging characteristic of mineral transformer oil.

Methods of test have been approved by Subcommittee IV for the detection and evaluation of the presence of combined but corrosive sulfur in mineral transformer oils. The method of test for the presence of free sulfur in transformer oil as revised by Subcommittee IV has been approved by Committee D-9.

Work has been continued on methods of test for evaluating the amount of dissolved and suspended water in oil. A method of test based on the extraction procedure has been drawn up and its accuracy and repeatability established on the basis of cooperative testing. A second type of test based on electrometric titration using the Karl Fischer reagent is still being investigated by the cooperating laboratories. It is expected that this investigation will be concluded during the next year.

The Standard Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin (D 877 - 49) has been limited in its application to oils of less than about 150 sec Saybolt Universal at 100 F. Oils of substantially higher viscosity (as for example oils with a viscosity of about 100 sec Saybolt Universal at 210 F) have been found to give widely erratic results with this testing procedure. The development of a testing method suitable for determining the dielectric strength of such oils is in progress.

During 1952 and 1953 other projects were in progress, including a revision of the Tentative Recommended Practice for the Purchase of Uninhibited Mineral Oil for Use in Transformers and Oil Circuit Breakers (D 1040 - 49 T) and the Tentative Method of Test for Steam Emulsion of Lubricating Oils (D 157 - 51 T). In addition, two new section activities have been initiated. Section C has been established for the study of methods suitable for the determination of metals which may be present in trace amounts in mineral oil. Section F has been established for the survey and study of tests for specific application to oils for electric cables.

The symposium activities of Subcommittee IV have been continued during 1952 and 1953. A symposium on the subject "Reclaiming and Inhibiting of

Used Insulating Oil" was held in June, 1952.[6] Another general symposium is being planned to be held in the fall of 1954.

Subcommittee V on Ceramic Products (R. W. Orr, chairman) is reviewing the Standard Methods of Testing Electrical Porcelain (D 116 – 44) for moderization and improvement. Work has been started toward the standardization of test methods and materials for internal ceramic insulators in electron tubes.

Subcommittee VI on Solid Filling and Treating Compound (W. R. Dohan, chairman). Drafts of proposed revisions of the Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 44) and of the Tentative Methods of Testing Hydrocarbon Waxes Used for Electrical Insulation (D 1168 – 51 T) have been submitted to letter ballot of the subcommittee.

Several sections are actively working on other projects as follows: Section E is preparing significance statements for Method D 176. Section F has made considerable progress on the preparation of methods of test for polymerizable embedding materials. These relatively new materials afford advantages over conventional materials used in the past, in heat resistance, shock resistance, and dielectric properties. In many cases the usual container may be dispensed with.

Subcommittee VII on Insulating Fabrics (R. W. Chadbourn, chairman) has proposed the Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 52 T) for adoption as standard and recommended its approval as an American standard by the ASA. The Section on Glass Fabrics has begun work on specifications for silicone varnished glass fabrics. It has also undertaken to develop test methods for silicone-rubber-coated fabrics.

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used for Electrical Insulation (D 295 – 52 T) were again reviewed during the year and a number of minor revisions made. A similar review of Tentative Specifications for Black Bias-Cut Varnished Cloth and Varnished Cloth Tape Used for Electrical Insulation (D 373 – 51 T) has been begun. Particular attention is being given to the dielectric strength requirements. A study of the available data on dielectric strength under 6 per cent elongation has been under way for some time with a view to possible revisions of existing requirements to meet changed conditions. The Section on Varnished Cloth has completed a study of the effect of electrode size and shape on the determination of power factor by the present method. It is also preparing specifications for oil for oil-packed tapes.

The subcommittee has approved extensive revisions in the Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 48) and Tentative Specifications for Flexible Treated Cotton and Rayon Sleeving Used in Electrical Insulation (D 372 – 47). These contain a complete reclassification of the materials involved, and enlargement of the scope of the specifications to include high-temperature or Class H insulations, as well as nylon and other materials not included now.

Tentative Methods of Testing Pressure-Sensitive Adhesive Tapes Used for Electric Insulation (D 1000 – 48 T) were reviewed during the year and a number of minor revisions are being recommended. Progress is under way on significance statements for these methods. The development of test methods for elastomeric pressure-sensitive tapes is progressing rapidly. Procedures for sampling, conditioning, and dielectric strength have been approved, and round-robin investigations are under way on several other test methods.

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[6] Issued as "Symposium on Insulating Oils—Fifth Series," issued as separate publication *ASTM STP No. 152.*

Subcommittee VIII on Insulating Papers (C. E. Peterson, chairman).—Revisions were made in 1952 in the Tentative Specifications for Absorbent Laminating Paper for Electrical Insulation (D 1080) and in the Tentative Methods of Sampling and Testing Untreated Papers Used for Electrical Insulation (D 202). Further revisions in Methods D 202 have been recommended for letter ballot by Committee D-9 to add four significance statements and a revision of the test method for bursting strength.

All sections have been active. Section A is developing test methods for chlorides, less than 50 ppm; acidity or alkalinity by potentiometric end point; and pH of unbuffered, neutral papers. Section B has completed a test method for dielectric strength of insulating papers and is currently engaged on a study of uniformity of absorbency of laminating papers of the type covered in Specifications D 1080. Section C completed the bursting strength test over 200 psi and has under consideration a test method for stretch of creped insulating papers. Section E has completed a series of significance statements for existing tests in Methods D 202 and, in addition to continuing this phase, is coordinating the work of this section with the current activities of their sections. Section F has completed specifications for insulating paper-interlayer type for letter ballot by Subcommittee VIII. Present activity involves the drafting of specifications for capacitor tissues. A task force will be set up for the study of an accelerated life test for capacitor tissues.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman).—Revision has been completed on Specifications D 351, as presented earlier in this report. Work is in progress on the Standard Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 - 49).

The subcommittee, in connection with its responsibilities for handling the American contacts with Committee on Mica of the International Standards Organization (ISO TC/45), has reviewed a number of documents received from the Indian Standards Committee, which handles the Secretariat. Some of the suggestions received were approved, and a task group was selected to investigate some of the debated points.

Round-robin tests are being continued on the new parellel-to-lamination test per Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 - 52 T), Appendix I, Method B. Preliminary results indicate that the cross-checks are satisfactory. When completed, it is hoped that these tests will verify and control the electrical quality of mica for capacitors.

In a general discussion of the current mica situation and special applications, it was brought out that significantly different performances of CL and SS ruby mica from different producing areas is evident at elevated temperatures, particularly with respect to dielectric strength and insulation resistance, and suitable test methods for such uses are desired.

Subcommittee XI on Significance of Test (J. H. Palmer, chairman).—Recommendations for the positioning of significance statements have been prepared and submitted to Committee D-9 for letter ballot.

Subcommittee XII on Electrical Tests (G. M. L. Sommerman, chairman).—Section A on resistivity is studying additional revisions of the Tentative Methods of Test for Electrical Resistance of Insulating Materials (D 257 - 52 T). Section C on dielectric loss has made extensive revisions to the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 47 T). A final draft will be distributed for subcommittee letter ballot. Section E on dielectric strength is revising the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 44). Section G on arc-resistance is preparing additions for the Tentative Method of Test for High-Voltage, Low-Current Arc Resistance of Solid Electrical Insulating Materials (D 495 – 48 T), covering high-current arc tests. Section H on electrodes has prepared a draft of a new tentative method on the measurement of dielectric properties of aviation fuels. This will be submitted to subcommittee letter ballot.

A new section designated K has been set up as a result of a request of Committee D-9 to study and prepare test methods for high-temperature performance of insulating materials.

Subcommittee XIV on Conditioning (A. C. Webber, chairman).—This Joint D-9, D-20 subcommittee prepared the Tentative Specifications for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 – 52 T), which replaced Specifications D 760 and D 761 and is the first enclosure specification based entirely on performance. Recent studies have shown it to be of real value to equipment producers, although further revisions are planned.

Active study has been initiated on the problems of accurate humidity measurement, particularly in enclosures and at high humidities. Satisfactory techniques and instrumentation for these measurements are not now available in ASTM methods.

Tentative Methods of Conditioning of Plastics and Electrical Insulating Materials for Testing (D 618 – 51 T) and Tentative Recommended Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions (D 1041 – 51) have been recommended to Sectional Committee C-59 for approval as American Standards by the ASA. Methods D 618 – 51 T are being recommended for adoption as standard as covered earlier in this report.

Subcommittee on Symposia (L. B. Schofield, chairman).—A Panel Discussion on methods of measuring dielectric constant and dissipation factor was held on March 26, 1953, with I. G. Easton as chairman, and Subcommittee XII as the sponsor.

The following new subcommittee chairmen have been appointed for their first term: O. E. Anderson—Subcommittee III; M. Guarnier—Subcommittee VII; and H. A. Anderson—Subcommittee VIII.

This report has been submitted to letter ballot of the committee, which consists of 179 members; 144 returned their ballots, of whom 123 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. H. SCOTT,
Chairman.

B. E. ELY,
Secretary.

REPORT OF COMMITTEE D-10

ON

SHIPPING CONTAINERS*

Committee D-10 on Shipping Containers held two meetings during the year: both in Chicago, Ill., on October 17, 1952, and on April 23, 1953.

The committee suffered serious loss through the death of Chairman T. A. Carlson and the resignation of Secretary J. H. Toulouse. A memorial resolution for Mr. Carlson has been prepared, an abstract of which appeared in the December, 1952 issue of the ASTM BULLETIN.

Officers elected at the Spring Meeting to fill the unexpired terms of chairman and secretary are:

Chairman, Gordon E. Falkenau.

Secretary, R. F. Uncles.

The membership consists of 98 voting members, composed of 34 producers, 39 consumers, and 25 general interest members.

All of the standards and tentatives for which Committee D-10 is responsible have been reviewed, and the necessary recommendations are included in this report.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards a Proposed Tentative Method of Test for Water Vapor Permeability of Packages by Cycle Method. This recommendation was accepted by the Standards Committee on April 16, 1953, and will appear in the 1953 Supplement to the Book of ASTM Standards, Part 7, bearing the designation D 1251 – 53 T.

NEW TENTATIVE

Committee D-10 recommends for publication as tentative the Method of Test for Water Vapor Permeability of Shipping Containers by Cycle Method, as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD

Committee D-10 recommends that the Tentative Methods of Testing Large Shipping Cases and Crates (**D 1083 – 50 T**) be approved without change for reference to letter ballot of the Society for adoption as standard.

CONTINUATION OF STANDARDS AND TENTATIVES WITHOUT REVISION

Committee D-10 recommends that the following standards, which have stood without revision for six or more years, be continued without revision:

Standard Methods of Test for:

Compression Test for Shipping Containers (D 642 – 47),

Drop Test for Shipping Containers (D 775 – 47), and

Shipping Containers in Revolving Hexagonal Drum (D 782 – 47).

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

Committee D-10 recommends that the following tentatives, which have stood without revision for two or more years, be retained as tentative:

Tentative Methods of Test For:

Vibration Testing for Shipping Containers (D 999 - 48 T), and
Pallets (D 1185 - 51 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Definitions of Terms (Edward Dahill, chairman) completed an additional list of definitions of terms to be included in the Standard Definitions of Terms Relating to Shipping Containers (D 996 - 50), which are now in process of letter ballot in Committee D-10. The subcommittee also completed a review of a proposed glossary of terms relating to interior packing, compiled by Subcommittee VI, which is also in process of committee letter ballot.

Subcommittee II on Methods of Testing (G. E. Falkenau, chairman) has been combined, for an indefinite period, with *Subcommittee IV on Performance Standards* (J. G. Turk, chairman) and *Subcommittee V on Correlation of Tests and Test Results* (R. C. McKee, chairman) for the purpose of conducting a coordinated program on factors affecting the results obtained from the standard test methods now being used in evaluating shipping containers. Four task groups have been organized to study the present drop test, revolving drum test, vibration test, and to develop a stacking test. The study of statistical methods is being continued with an augmented task group. Decision was reached to drop further studies in the development of a puncture resistance test of multiwall paper shipping sacks.

Subcommittee VI on Interior Packing (S. L. Swenson, chairman) has been working on a bibliography and abstract of cushioning studies, which is expected to be completed in the near future. A proposed load deflection method for determining energy absorption of cushioning materials has been reviewed, and check determinations will be made during the next few months. The measurement of compression set of cushioning materials has been considered, and tests will be made to develop data for a constant load method.

This report has been submitted to letter ballot of the committee, which consists of 98 members; 65 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

EARL STIVERS,
Acting Chairman.

L. C. GILBERT,
Acting Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials, and twelve of its subcommittees, held meetings in Detroit, Mich., on March 4 to 6, 1953, during ASTM Committee Week. In addition, some of the subcommittees have held separate meetings during the year; and the SAE-ASTM Technical Committee on Automotive Rubber, with its various sections, has met on a quarterly basis.

Progress has been made during the year in the establishment of standard test formulations and lots of compounding materials. The recipes and procedures for compounding and mixing are now included in ASTM Tentative Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 - 52 T), which also includes a list of standard ingredients. It is hoped all may eventually be made available from a standard source such as the National Bureau of Standards. During the year the Bureau has added one new standard ingredient to the list which it was previously prepared to supply. This is N.B.S. Standard Sample No. 377, phenyl beta naphthylamine. There is still no standard source for the rubbers nor for SRF black, acetylene black, Silene EF and whiting. Further work by the Bureau on this project has not been undertaken because of limitations in funds and personnel.

The committee has continued its activity in the work of Technical Committee 45 on Rubber of the International Organization for Standardization (ISO). Several of the subcommittees have investigated and reported on proposals under study in the ISO committee. Committee D-11 will be represented at the fifth meeting of ISO/TC 45 in Paris, June 15 to 23, by R. D. Stiehler, National Bureau of Standards; J. R. Britt, U. S. Department of the Navy, Bureau of Ships; Irving Kahn, U. S. Department of the Army, Office of the Chief of Ordnance; J. R. Kerscher, Goodyear Tire & Rubber Co.; and J. C. Montermoso, U. S. Department of the Army, Office of the Quartermaster General.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

The following recommendations presented to the Society through the Administrative Committee on Standards were accepted on November 13, 1952:

Revision of Tentative Methods of:

Testing Automotive Hydraulic Brake Hose (D 571 - 51 T),

Test for Compression Fatigue of Vulcanized Rubber (D 623 - 41 T),

Testing Asphalt Composition Battery Containers (D 639 - 51 T),

Testing Compression Asbestos Sheet Packing (D 733 - 50 T),

Testing Rubber-Coated Fabrics (D 751 - 46 T),

Test for Resistance of Vulcanized Rubber or Synthetic Elastomers to Cut Growth by the Use of the Ross Flexing Machine (D 1052 - 49 T),

Measuring Low-Tempterature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus (D 1053 - 52 T).

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28- July 3, 1953.

Revision of Tentative Specifications for:

Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 - 52 T).

Revision of Tentative Specifications and Methods of Test for:

Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex (D 1076 - 52 T).

Withdrawal of Standard Methods of:

Sampling and Testing Latices of Natural Rubber and Synthetic Rubbers (D 640 - 44).

The revised tentatives appear in the 1952 Book of ASTM Standards, Part 6, as well as in the 1952 Compilation of ASTM Standards on Rubber and Rubber-like Materials.

The revision of the tentative methods of testing Automotive Hydraulic Brake Hose, D 571, provides for the addition of the $\frac{3}{16}$-in. size of hose and a change in the cold test procedure from -40 F to -65 F.

In the Test for Compression Fatigue of Vulcanized Rubber, D 623, the revision clarifies some test details and provides for the use of molded as well as die-cut specimens. The revision of the Tentative Methods of Testing Asphalt Composition Battery Containers, D 639, adds a procedure for determining the susceptibility of a container to fracture by means of a drop ball impact test. The change in the Methods of Testing Compressed Asbestos Sheet Packing, D 733, defines more precisely the speed of the testing machine. The revision in the Tests of Rubber-Coated Fabrics, D 751, provides for a definite distance of separation of 3 in. in the adhesion test. In the Test for Resistance of Vulcanized Rubber or Synthetic Elastomers to Cut Growth by the Use of the Ross Flexing Machine, D 1052, the size of the piercing tool and holder are defined more accurately than formerly. In the revision of the Test for Low-Temperature Stiffening by the Gehman Torsional Apparatus, D 1053, changes were made clarifying the method of calibration of torsion wire and adding the calibration as a new section of the method instead of handling by a Note. A new section was also added giving the equations and method for calculating modulus of rigidity of the test piece.

In the revision of the Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications, D 735, changes were made in Table I covering Type R compounds, non-oil resistant, Table III Type S, Class SB, oil resistant synthetic rubber compounds, and Table IV Type S, Class SC, oil resistant synthetic rubber compounds, adding intermediate grades which the committee feels are needed. A new Table VI covering polyacrylic rubber resistant to heat and petroleum hydrocarbons was added. In addition, compression set as a basic requirement in Table V on silicone rubbers was removed as this is considered adequately covered by Suffix B in the Table. A number of editorial changes were also made in the specifications, including the specific exclusion of insulated wire and cable compounds from the scope of these specifications.

The revision of the Tentative Specifications and Methods of Test for Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex, D 1076, reduces the requirement for mechanical stability from 600 sec minimum to 400 sec minimum, changes the dimensional tolerances on equipment for the mechanical stability test, as well as some details of the procedure and the definition of the end point. These changes resulted, to a considerable extent, from discussions in ISO/TC 45. The withdrawal of the Standard Methods of Sampling and Testing Rubber Latices of Natural Rubber and Synthetic Rubbers, D 640, was recommended because this standard has been superseded by similar provisions in method D 1076.

NEW TENTATIVES

Committee D-11 recommends the acceptance for publication of the following two new tentatives as appended hereto:[1a]

Tentative Specifications for Non-Rigid Thermoplastic Compounds for Automotive and Aeronautical Applications.—These tentative specifications have been under consideration for several years in the SAE-ASTM Technical Committee on Automotive Rubber, which has now recommended their publication by both ASTM and SAE. Committee D-11 concurs in this recommendation and believes that these specifications, which are prepared in form similar to the corresponding specifications for rubber compounds, will fill a long-felt need in the automotive industry.

Tentative Methods for Determining Harmful Dirt in Crude Natural Rubber.—These tentative methods have been developed by Subcommittee XII on Crude Natural Rubber in connection with its work in developing standard test procedures for the evaluation of this material. The presence of foreign matter has always been one of the principal sources of difficulty in the control of crude rubber quality. Ordinary inspection methods fail to evaluate this defect adequately. The two procedures recommended in this proposed tentative have been thoroughly investigated by the subcommittee and found to be satisfactory when used as outlined in the scope. These methods are also being suggested to the ISO/TC 45 for their consideration. It is felt that their approval will be very helpful in controlling one of the most troublesome defects found in crude natural rubber.

REVISION OF TENTATIVES

The committee recommends that nine of the present tentatives be revised as indicated and continued as tentative:

Tentative Methods of Test for Compression Set of Vulcanized Rubber (**D 395 – 52 T**:[1]

Section 8: Heat Treatment.—In Paragraph (*c*) delete the portion of the second paragraph which reads "Before inserting the test specimen in the compression device, the compression device shall be preheated, etc." and ends with the words "for a period of 20 min. before being replaced in the air oven." Substitute for the above the following sentence: "The test specimen shall be at room temperature when inserted in the compression device." This change eliminates the requirement for preheating the compression device before loading in both Methods A and B. Investigation by the subcommittee has proved that preheating is neither necessary nor advisable.

Tentative Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (**D 865 – 52 T**)[1]:

Section 5: Test Specimens.—Change Paragraph (*b*) to read as follows:

> "The cross-sectional dimensions for calculating the physical properties shall be measured prior to exposure in the aging chamber. Gage lines used for measuring elongations shall be applied after the specimens have been aged. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other."

The committee has investigated the practice of placing elongation bench marks on test specimens both before and after exposure to the elevated temperature. Marking before exposure often results in loss of the marks in handling or adverse action of the ink material on the rubber. Application of the marks after exposure would be objectionable only in the event of substantial change in dimensions during the exposure time. The work of the subcommittee on specimens using various polymers has indicated that these dimensional changes are considerably less than experimental error in the method except when specimens are actually melted or approaching a melted condition, in which case the test would not ordinarily be run. The subcommittee advises that in most cases marking after exposure is preferable and is in line with the practice usually followed.

[1a] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 6.

[1] 1952 Book of ASTM Standards, Part 6.

Tentative Methods of Testing Compressed Asbestos Sheet Packing (**D 733 - 52 T**).[1] The following changes are recommended:

Section 5: Measurement of Thickness.—Change the last sentence by deleting the words "around the borders of the article" and substitute the phrase "over the area of the article." This change is intended to permit measurements elsewhere than at the border if the center of the article happens to be accessible.

Section 8: Compressibility and Recovery.—Change the major load value of 250 lb to the value of 245 lb in order to be consistent with Method D 1147, the Compressibility Test Method.

Section 10: Immersion Tests.—In Paragraph (*b*) delete the second and last sentences referring to compressibility after immersion.

Tentative Method of Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (**D 927 - 52 T**):[1]—As a result of work done at the National Bureau of Standards on the influence of variation in rotors, dies, and rate of shear on the Mooney Viscosity, the committee recommends the following revisions:

Delete Fig. 1 showing line drawing of

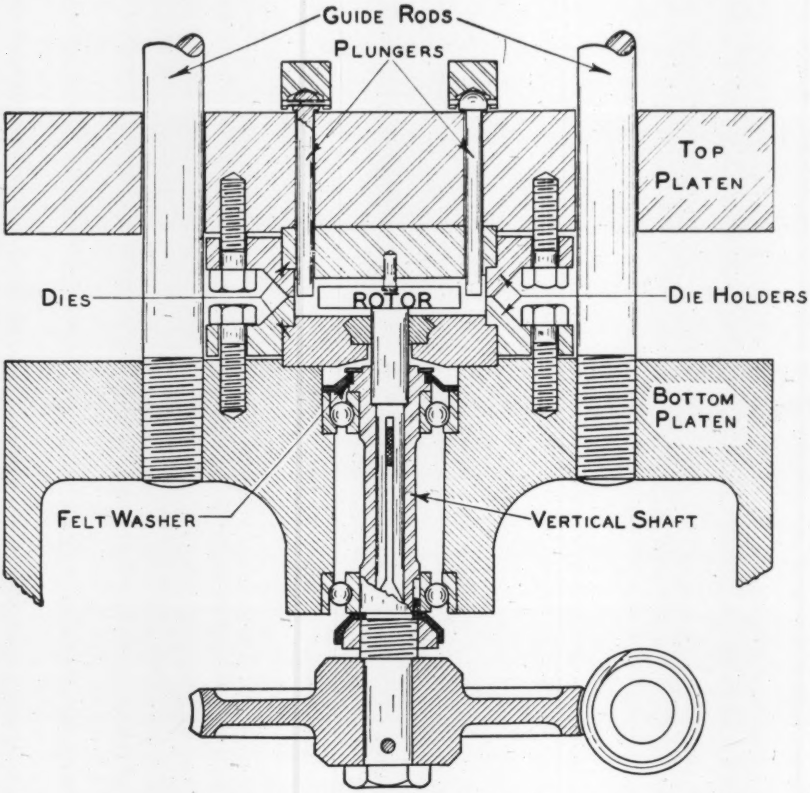

FIG. 1—Shearing Disk Viscometer.

the older type of Mooney Viscometer and substitute a similar drawing of the NBS Model.

Section 2: Apparatus.—In Paragraph (*a*) delete the word "suitably" from the last sentence and add "The serrations on the die shall be formed by cutting square grooves $\frac{1}{32}$ in. wide with a uniform depth of not less than 0.010 in. nor more than 0.015 in. The grooves forming the serrations on the die holder shall be vertical and shall be cut on $\frac{1}{16}$-in. centers. The serrations on the dies shall be formed by cutting 2 sets of grooves at right angles to each other." Add a new paragraph to read as follows:

> "The pin in the center of the serrated face of the upper die shall be $\frac{1}{8}$ in. in diameter and shall extend not more than 0.123 in. nor less than 0.121 in. beyond the serrated face of the die."

Revise Paragraph (*b*) to read as follows: "(*b*) The die shall be an integral part of, or mounted on platens equipped with, a heating device and controls capable of maintaining the die cavity at a specified equilibrium test temperature with a tolerance of ±1 F."

On item 3 in the note under Paragraph (*b*), change the first sentence to read: "In cases in which the viscometer has not been equipped with thermocouples as specified in the Tentative Method of Test for Curing Characteristics of Vulcanizable Mixtures During Heating by the Shearing Disk Viscometer (D 1077 – 49 T), a thermocouple may be inserted, etc."

Revise Paragraph (*c*) to read as follows: "(*c*) The dimensions of the rotor head, measured from the tops of the serrations, shall be 1.500 ± 0.001 in. in diameter and 0.218 ± 0.001 in. in thickness. The rotor head shall also be serrated to prevent slippage. The serrations on the face of the rotor shall conform to the requirements given in Paragraph (*a*) for the serrations on the die. The serrations on the edge of the rotor shall conform to the requirements given in Paragraph (*a*) for the serrations on the die holders. The rotor head shall be securely mounted by means of a suitable shaft so that the horizontal center line of the rotor and die cavity shall coincide within plus or minus 0.005 in. The eccentricity or runout of the rotor shaft with the rotor in the machine shall not be greater than 0.0005 in. It shall be permissible to use a small rotor having a head diameter of 1.200 ± 0.001 in. in place of the large rotor when testing a stock having a viscosity greater than 200-ML units. Except for the diameter, the small rotor shall conform to the dimensions for the large rotor. The counterbore (or spot face) in the top of the rotor shaft shall be 0.027 in. plus 0.000 in. minus 0.001 in. deep measured from the top of the serrated surface. The shoulder of the rotor shaft shall be 0.4070 in. plus 0.0000 in. minus 0.0005 in. in diameter. It shall bear on the vertical shaft and not on the lower die (see Fig. 1)."

Tentative Method of Test for Impact Resilience and Penetration of Rubber by the Rebound Pendulum (**D 1054 – 49 T**):[1]

Section 5: Test Conditions.—In Paragraphs (*a*) and (*b*) change the specified testing temperature from 82 ± 2 F to 73.4 ± 2 F. This change is necessary to bring the test temperature in line with the presently accepted standard.

Tentative Specifications and Methods of Test for Latex Foam Rubbers (**D 1055 – 52 T**):[1]

Section 4: Grades of Latex Foam Rubbers.—The following changes are recommended in the list of Suffix Letters: (1) Suffix Letter A should be dropped since no oven aging test is used other than those under basic requirements. (2) Suffix Letter B should be dropped since no compression set test other than the basic requirement is approved. (3) Suffix FF should be removed since at present no test other than the one low temperature test is used. If one is to be added later, it probably would be designated F_1 or F_2. (4) Under M change to read "Inflammability test required.*" (5) Under N change to read "Repeated impact test required.*"

Tentative Specifications and Methods of Test for Sponge and Expanded Cellular Rubber Products (**D 1056 – 52 T**).[1]—This revision incorporates a change in the water absorption test which has been requested by the Technical Committee on Automotive Rubber and is recommended by Subcommittee XXII.

Section 34: Test Specimens.—Replace the present paragraph with the following:

> "Standard test specimens approximately ½ in. in thickness and 4 sq. in. in area shall be used for this test. Round specimens are preferable. The test specimens shall have the natural skin on the top and bottom surfaces of the disks."

Section 35: Procedure.—Replace the present paragraph with the following:

> "Specimens shall be submerged in distilled water at room temperature (65 to 95 F) 2 in. below the surface of the water and subjected to a vacuum of 25 in. of mercury for 3 min. The vacuum is released, and specimen still submerged is allowed to stand for three min at atmospheric pressure. The specimen is then removed, blotted dry, and the percentage change in weight is calculated."

Tables I, II, and III.—Under Suffix L in the tables, change the present requirement for maximum per cent water absorption from the present figure of 10 per cent to a maximum of 5 per cent.

Section 5(b).—In the table of Suffix Letters, delete Suffix Letter A and Suffix FF as in Specifications D 1055. Change Suffix B to read as follows: "Compression set test required with values as specified in Table III." Remove asterisk at end of sentence after III in Suffix L. Change Suffix M to read "Inflammability test required.*" Change Suffix N to read "Repeated impact test required.*"

Tentative Method of Test for Compressibility and Recovery of Gasket Materials (**D 1147 – 51 T**).[1]—The following revisions are recommended in order to make the test method more definite and to clarify ambiguities:

Section 2: Apparatus.—In Paragraph (*b*) change last sentence to read "Penetrator diameters for various types of gasket materials are as shown in Table I, unless otherwise specified." In Paragraph (*d*) change last sentence to read: "Preloads for various types of gasket materials are as shown in Table I, unless otherwise specified." Change the next to the last sentence in Paragraph (*e*) to read: "The major load shall be in addition to the specified preload." Change the last sentence to read: "Major loads for various types of gasket materials are as shown in Table I, unless otherwise specified."

Table I.—Omit the word "Recommended" from the title and change values in "Major Load" column to read respectively 245 lb, 79 lb, 45 lb, 49 lb, 49 lb, 99 lb, 49 lb, 49 lb. Add a final column entitled "Sum of Major Load and Preload" giving values as at present in column entitled "Major Load."

Section 4: Conditioning of Specimens.—Change last sentence to read as follows: "Conditioning procedures for various types of gasket materials are as shown in Table I, unless otherwise specified."

Tentative Methods of Testing Adhesives for Brake Lining and Other Friction Materials (**D 1205 – 52 T**).[1]—The committee recommends the following revisions as refinements in the tentative methods:

Section 4(b): Elevated Temperature.—After the first sentence insert the following new sentence: "The rate of heating may be a matter of agreement between buyer and seller."

Section 5: Apparatus.—At the end of the first sentence following the words "at a rate of 0.4 in. per min" add the phrase "or at a loading rate not to exceed 1000 lb per sec." At the end of Section 5 add two additional sentences as follows:

"Alignment of the fixture should be parallel. Caution should be exercised to avoid preliminary load prior to start of test."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Method of Air Pressure Heat Test of Vulcanized Rubber (**D 454 – 52**),[1]

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (**D 572 – 52**),[1] and

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (**D 573 – 52**).[1]

The committee recommends for immediate adoption the same revisions in the corresponding paragraphs of these three standard methods as recommended above in the Tentative Method of Heat Aging by the Test Tube Method (D 865 – 52 T). The reasons are the same in all cases and it is considered desirable to have the changes published simultaneously. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction, other than those mentioned in this report, be continued as tentative. Most of these are being reviewed by the various subcommittees, and the results of this work will be presented to the Society as it is completed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Mr. J. F. Kerscher, Goodyear Tire and Rubber Co., has been appointed chairman of the new subcommittee authorized to develop specifications for rubber compounds for general application similar to the SAE-ASTM Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (ASTM Designation D 735), which have been used so successfully in the automotive and aeronautical fields.

Numerous interesting projects are engaging the attention of various subcommittees of Committee D-11. It is possible here to mention only a few of these, but it should be noted that other subcommittees have a large amount of work in progress which will be reported in due course.

Subcommittee V on Insulated Wire and Cable has completed drafts of proposed specifications for insulation compounds based on butyl rubber and polyethylene. Both of these have been in the formative stage for some time and will be presented to the Society when agreement is reached. Meanwhile progress is being made on the master specification covering construction details of insulated wire and cable used for the distribution of electrical energy which was mentioned in a previous report.

Subcommittee XII on Crude Natural Rubber has been giving attention to methods for defining and measuring the vulcanization characteristics of crude rubber in addition to its work on the quantitative determination of dirt, copper, and manganese in rubber. The most important property of natural rubber for technical classification is its vulcanization behavior. Variations in this characteristic cause considerable difficulty during manufacture. The principal control of this property in the past has been obtained by determination of the strain or modulus of a standard test vulcanizate cured in a standard way. The subcommittee feels that an adequate evaluation of the measurement of vulcanization will require not only the present measurements, but in addition, measurement of the time of incipient cure and rate of cure, preferably determined by the change in Mooney viscosity of a test compound as a function of time at vulcanizing temperatures.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A number of round-robin test programs are under way in various subcommittees. Subcommittee XV has received a report from a section working on the correlation of oven and shelf aging tests. Aging periods of two, three, and four years, both in the United States and in Liberia, have been covered and the next series of tests will be made at the end of eight years. The extensive data obtained are to be analyzed by Subcommittee XXVIII on Statistical Quality Control. An extensive program on the measurement of the resistance of rubber to ozone aging is being carried out by a section functioning in both the Technical Committee on Automotive Rubber and Subcommittee XV. A large amount of data has already been reported and the work is still in progress. Seven rubber compounds of different types are involved and correlations are being obtained between outdoor exposure in various locations and tests in ozone cabinets. Another testing program is in progress in the Technical Committee on Automotive Rubber on static exposure testing. This program is in its second year. Six different samples are being exposed in eighteen different locations. A large amount of data on rate of cracking at different seasons in different locations is being evaluated in the effort to improve present methods of outdoor weather aging and to correlate results of outdoor aging with laboratory accelerated tests.

Subcommittee XX on Adhesion Tests is planning revisions of the Tentative Methods of Test for Adhesion of Vulcanized Rubber to Metal, D 429, and has outlined an extensive round-robin test program to evaluate these proposed revisions. Before this program is started it is being carefully planned and reviewed by Subcommittee XXVIII on Statistical Quality Control to make certain that adequate data will be obtained on which to base sound conclusions as to the relative merits and reproducibility of both the proposed and the existing methods. Subcommittee XXI has completed a round-robin test program of brake lining adhesive, tested according to Methods D 1205 – 52 T, in order to evaluate the reliability of these methods. The results which have been reported show that the methods are generally satisfactory, although a few minor changes are indicated and will be recommended in due course.

Subcommittee XXV is considering a draft of a proposed new tentative known as the T-R (Temperature-Retraction) Test Procedure. This method of test describes a procedure for determining the retraction characteristics of rubber and rubber-like materials caused by a rise in temperature after the materials have been frozen in an elongated condition at sufficiently low temperatures to cause them to lose practically all of their elastic properties. The retraction values obtained give an over-all picture of low-temperature behavior including the effect of crystallization. It is expected that this proposed method will be presented to the Society for publication in the near future.

This report has been submitted to letter ballot of the committee, which consists of 211 members; 139 members returned their ballots, of whom 138 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

REPORT OF COMMITTEE D-12

ON

SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year on March 16 and 17, 1953, in New York, N. Y.

During the year William G. Morse retired, and has resigned as Chairman of Subcommittee G-2 on Nomenclature and Definitions. This vacancy has not yet been filled. A. M. Schwartz has been appointed to replace Mr. Morse on Subcommittee G-1, Advisory.

At the present time the committee consists of 115 members, of whom 58 are classified as producers, 35 as consumers, and 22 as general interest members, with 6 consulting members.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information the proposed Method of Aerated Total Immersion Corrosion Test for Metal Cleaners, as appended hereto.[1]

NEW TENTATIVES

The committee recommends that the following four methods of test be accepted for publication as tentative:[2]

Tentative Methods of Test for:

Rinsing Properties of Metal Cleaners,
Buffering Action of Metal Cleaners,
Total Immersion Corrosion Test for Soak Tank Metal Cleaners, and
Analysis of Sodium Bicarbonate.

A revision of the proposed Tentative Method of Test for Buffering Action of Metal Cleaners is recommended, as follows:

Section 2(c).—Revise to read as follows:

(*c*) *Buffer Index.*—The milliliters of 0.5 *N* hydrochloric acid required to decrease the initial pH of the solution either 1 pH unit or some definite change in pH as agreed upon by the purchaser and producer. Examples of change in pH ranges are as follows:

(*1*) From initial pH of a solution to pH of 12.0,
(*2*) From pH 12.0 (or initial pH if between 11 and 12) down to pH 11.0, and
(*3*) From pH 11.0 down to pH 10.0

The proposed Tentative Method o Test for Analysis of Sodium Bicarbonate is in effect a tentative revision of the Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (**D 501 – 49**),[3] and is intended to be added eventually to the standard methods.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following three tentatives be approved for reference to letter ballot of the Society for adoption as standard, without revision:

Tentative Specifications for:

Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer) (D 1111 – 51 T),[3] and
Solid Soap for Low-Temperature Washing (Low and Medium Titer) (D 1112 – 51 T).[3]

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] See p. 438.

[2] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

[3] 1952 Book of ASTM Standards, Part 7.

Tentative Method of Test for:

Foaming Properties of Surface-Active Agents (D 1173 - 51 T).[3]

Revision of Standard and Reversion to Tentative

The committee recommends that the Standard Specifications for Milled Toilet Soap (**D 455 - 48**)[3] be revised as follows and reverted to tentative:

Section 3.—Revise the second, fourth, and last chemical requirements to provide for an increase in the permissible value, to read as follows:

Sum of free alkali, total matter insoluble in alcohol, and sodium chloride, max., per cent	3.0
Matter insoluble in water, max., per cent	1.0
Anhydrous soap content, min., per cent	81.0

Tentative Continued Without Revision

The committee recommends that the Tentative Method of Test for pH of Aqueous Solutions of Soaps and Detergents (**D 1172 - 51 T**) be continued as tentative without revision.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

Activities of Subcommittees

Subcommittee T-1 on Soap Analysis (E. W. Blank, chairman).—This subcommittee, which functions jointly with a committee of the American Oil Chemists' Society, has completed cooperative tests on a method for the determination of copper in soap. This method has been forwarded to the Uniform Methods Committee of the A.O.C.S. and is under consideration for acceptance as a tentative method. A task group is being organized to develop a procedure for the determination of the color of bar soaps. Methods for the determination of sodium carboxymethyl cellulose and P_2O_5 in soaps are being studied.

Subcommittee T-2 on Analysis of Synthetic Detergents (R. Bernstein, chairman) has prepared a bibliography covering 61 methods of analysis for synthetic detergents. Approximately 15 current references are being added and the bibliography will be submitted for publication.[5] The subcommittee is now engaged in the preparation of a scheme for qualitative analysis of synthetic detergents.

Subcommittee T-4 on Analysis of Alkaline Detergents (W. H. Koch, chairman) has prepared the Tentative Method of Test for Analysis of Sodium Bicarbonate, which is appended to this report.[6] Methods for the analysis of sodium orthosilicate and sodium polyphosphates are being investigated. Specifications for anhydrous trisodium phosphate and anhydrous sodium metasilicate are to be studied.

Subcommittee T-5 on Physical Testing (A. M. Schwartz, chairman).—Task groups have been appointed to investigate methods of test for the determination of redeposition of soil on textiles and wetting of textiles.

Subcommittee T-6 on Metal Cleaning (J. C. Harris, chairman) has prepared a proposed Method of Aerated Total Immersion Corrosion Test for Metal Cleaners, which is appended as information.[1]

The subcommittee is engaged in the preparation of a laboratory guide to the evaluation of metal cleaners. This includes recommendations for methods of measurement of residual soil, a classification of typical soils, and other details. A task committee, consisting of

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] Issued as separate publication *ASTM STP No. 150.*

[6] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

H. Kafarski (chairman), A. Balden, and F. Chase, has been appointed to study methods of application of metal cleaners in the automotive industry.

Subcommittee S-1 on Specifications for Soap (F. Krassner, chairman) recommended changes in specifications as detailed earlier in this report and is continuing its general work on soap specifications.

Subcommittee S-4 on Specifications for Alkaline Detergents (M. F. Graham, chairman) is engaged in the development of specifications for sodium orthosilicate. Future plans include revision of specifications for soda ash and development of a specification for anhydrous sodium metasilicate.

Subcommittee G-1, Advisory (J. C. Harris, chairman) recommended revision of annual meeting programs to provide for consecutive meetings of subcommittees, in order to eliminate program conflicts. It further recommended revision of the membership lists of the various subcommittees with a canvass of the membership, to determine those in a position to participate actively. It also recommended that Subcommittee T-2 establish liaison with Committee E-13 on Absorption Spectroscopy, on infrared analysis.

Subcommittee G-5 on Program Planning (R. B. Mitchell, chairman) prepared the program presented at the meeting of the committee held on March 16 and 17, 1953, in New York, N. Y., which included, in addition to regular committee activities, presentation of the following technical papers:

Metal Cleaning, by William Stericker,
The Effects of Detergents on Sewage Treatment Processes, by Raymond Manganelli,[7]
Analysis of Hard Water Soaps: The Direct Determination of Active Sulfonate in Mixtures of Soap and Nacconol RB-85, by L. F. Hoyt and J. E. Walter,[7]
A Multi-Surface Cleaning Test, by M. N. Fineman,[7]
Asphalt Tile Cleaner Specifications, by A. C. Zettlemoyer, [7] and
Dry Cleaning, by G. P. Fulton.[7]

This report has been submitted to letter ballot of the committee, which consists of 115 members; 79 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

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[7] Published in ASTM BULLETIN, No. 192, September, 1953, p. 49 (TP139). Also issued as a separate reprint.

PROPOSED METHOD OF AERATED TOTAL IMMERSION CORROSION TEST FOR METAL CLEANERS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method of test is intended as a means of determining the corrosive effects of "metal cleaners for use in mechanical equipment," on all metals other than aluminum and its alloys, under conditions of aeration and total immersion, by quantitative measurement of weight change or by qualitative visual determination of change. The test is designed for the determination of the effects of the cleaner on metals being cleaned and is not for determination of the life of the cleaner or of the containing equipment.

(*b*) When the test is used to assist in the choice of material for a specific use, the test conditions should simulate the conditions of use as closely as practicable.

(*c*) Where no further processing subsequent to cleaning is indicated, a test for the effect of residual cleaner on the corrosion behavior of the material may be required.

Apparatus

2. (*a*) *Test Tubes* fitted with a sintered-glass diffusion disc at the bottom, connected to a tubulation, for the purpose of saturating the solutions under test with air. The section containing the solution under test shall be 58 mm in diameter by 300 mm in length, and shall have a capacity of about 300 ml. The tubes shall be chosen so that the specimens will remain fully immersed during the test, the ratio of area of immersed metal to the volume of the solution will be as prescribed in Section 6 (*a*), and sufficient space will be provided for foam formation.

(*b*) *Source of Filtered Low-Pressure Air*, for aerating the test solution. The air used should be bubbled through sodium hydroxide solution (30 g NaOH per liter), maintained at room temperature, in order to remove carbon dioxide, and then through water, at or near the temperature of test, in order to reduce evaporation in the test cell.

(*c*) *Condensers*, Allihn-type, having jackets 200 to 250 mm in length. Condensers are not required for tests run at room temperature.

(*d*) *Stoppers or Joints.*—The connection between test tube and condenser optionally may be either standard-taper joints or rubber stoppers preboiled in aqueous caustic soda to remove free sulfur and boiled in a sufficient number of changes of distilled water until neutral.

(*e*) *Constant-Temperature Device.*—Any suitable means may be employed for maintaining the solutions actively at the boiling point for tests conducted at that temperature. For tests made at other temperatures, a suitable constant-temperature bath shall be provided to maintain the required temperature within ±2 F (1 C). For control testing at room temperature, it is suggested

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-12 on Soaps and Other Detergents.

[2] Published as information, June, 1953

that the solution be maintained at 95 ± 2 F (35 ± 1 C), which, being slightly above most room temperatures, is easy to maintain by heat input.

Apparatus for Residual Cleaner Corrosion Test

3. (*a*) *Coating Apparatus* for producing a uniform film of cleaner on the surface of the test specimens.[3]

(*b*) *Humidity Chamber* for exposing the contaminated specimens at a temperature of 95 F (35 C) and 90 per cent relative humidity.

Test Specimens

4. (*a*) *Selection of Samples.*—The samples selected for corrosion testing shall be identical in composition, metallurgy, and surface finishing with the metal at the stage where cleaning will be applied in practice.

(*b*) *Size and Number of Specimens.*—The test specimens shall have an area between 0.300 and 0.375 sq dm and a length not to exceed 200 mm. (A specimen 18 mm by 85 mm by 1 mm in thickness would have an area of 0.327 sq dm.) At least two, and preferably four, replicates shall be tested in each concentration of cleaner solution prescribed in Section 6 (*b*). The number of replicates under test shall be reported.

Preparation of Test Specimens

5. Immediately prior to their use in the test, clean the test specimens as directed in Section 4 of the Tentative Method of Test for Total Immersion Corrosion Test for Soak Tank Metal Cleaners (ASTM Designation: D 1280).[4]

Test Conditions

6. (*a*) *Ratio of Area of Immersed Metal to Volume of Solution.*—The ratio of the area of the immersed metal to the volume of solution shall be less than 1.5 sq dm per liter of solution. (A specimen 18 by 85 mm by 1 mm in thickness would require 220 ml of solution, and a specimen having an area of 0.375 sq dm would require 250 ml of solution.) Use fresh solution for each set of replicates, and record the ratio used.

(*b*) *Solution Concentration:*

(*1*) In the absence of the manufacturer's recommendations, test the specimens in solutions of the cleaner of concentrations of 0.25, 0.5, and 2.0 per cent by weight, made up from stock solutions which shall be freshly prepared in 2000-g quantities, according to the manufacturer's recommended procedure. Boil the solutions for 30 min, unless otherwise specified. In case the cleaner is not soluble to the extent noted in the stock solutions indicated, record this fact, but nevertheless continue the test with the specified total amounts of cleaner present in the test tubes. A blank test of either two or four replicates shall be made in freshly boiled distilled water.

(*2*) When the manufacturer's recommendations are available, make the test at the following relative concentrations (based on the average concentrations suggested), recording the percentages these represent:

One half the concentration recommended,
The concentration recommended and
Twice the concentration recommended.

Make a blank test of either two or four replicates in freshly boiled distilled water, submitting them otherwise to exactly the same procedure followed for the specimens immersed in cleaner solution.

(*c*) *Water.*—The water used in preparing the water-soluble cleaner solutions shall be freshly boiled distilled water.

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[3] The Fisher-Payne Dip Coater has been found satisfactory for this purpose.

[4] 1953 Supplement to Book of ASTM Standards, Part 7.

(*d*) *Temperature.*—When the manufacturer's recommendations are available, make the test at 20 ± 2 F (11 ± 1 C) below and 20 ± 2 F (11 ± 1 C) above the average recommended temperature, or at 180 ± 2 F (82 ± 1 C) if it is lower, the temperature being recorded in any case. In the absence of the manufacturer's recommendations, make the test at 180 ± 2 F (82 ± 1 C).

(*e*) *Aeration.*—Aerate the solution by passing air through the solution at the rate of 200 ml per liter per min. The volume of air should be measured and controlled as accurately as possible, preferably within plus or minus 10 per cent. For 250 ml of solution, 50 ml of air per minute would be required.

Procedure for Quantitative Weight Loss Test

7. Conduct the quantitative weight loss test as directed in Section 6 of Method D 1280.

Procedure for Qualitative Surface Corrosion Test

8. Proceed as for the quantitative weight loss test, except that drying and weighing the specimens before the test shall be omitted, and the removal of corrosion products may be dispensed with unless the corrosion is severe enough to obscure observation.

Procedure for Residual Cleaner Corrosion Test

9. Conduct the residual cleaner corrosion test as directed in Section 8 of Method D 1280.

Report

10. (*a*) *Quantitative Weight Loss Test.*—Report the following data for each test performed:

(*1*) Temperature,

(*2*) Specimens, size and number,

(*3*) Cleaner concentrations,

(*4*) Metal, alloy, surface treatment, and metallurgical state,

(*5*) Ratio of surface area to volume of solution,

(*6*) Aeration rate, in milliliters of air per liter of solution per minute,

(*7*) Type of cleaner and other conditions of test peculiar to type,

(*8*) Weight loss, in milligrams per square decimeter per hour,

(*9*) Range in weight loss values, and

(*10*) Appearance before and after removal of corrosion products, with regard to the following:

- Discoloration,
- Dulling,
- Etching,
- Presence of accretions and relative amounts and areas,
- Type of pitting—wide, medium, or narrow, and
- Presence of selective or localized attack.

(*b*) *Qualitative Surface Corrosion Test.*—Report the same information as for the quantitative weight loss test (Paragraph (*a*)), except to omit Items (*8*) and (*9*).

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS[*]

Committee D-13 on Textile Materials held two 3-day meetings during the year, in October, 1952, and March, 1953, both of them in New York, N. Y. The October meeting featured a Symposium on Thickness and Compressibility of Textiles,[1] at which papers presented by H. F. Schiefer, Herman Bogaty, and E. R. Frederick were the basis for discussion. The General Session at the March meeting was devoted to the presentation of the 1953 (fourth) Award of the Harold DeWitt Smith Memorial Medal to Prof. Herbert J. Ball, honorary chairman of Committee D-13 and Past President of the Society.

A survey of activities of the Task Groups in Committee D-13 revealed that there are currently 80 groups, each working on a different subject, with a total of 215 individuals participating in the work.

A new subcommittee to develop standard test methods for warp knit fabrics was authorized by the Advisory Committee in March as a result of industry conferences at which representatives of the National Federation of Textiles, Underwear Institute, dyers of warp knit fabrics, fiber producers, and other organizations decided to turn to Committee D-13 for work in this field. Several Task Groups started work at once and the subcommittee is being organized.

W. M. Scott, A. F. Tesi, and W. D. Appel of Committee D-13 served as American representatives at meetings of the International Organization for Standardization Subcommittee on Colorfastness Test Methods in New York on November 10, 11, and 12, 1952. Twenty-four methods were agreed upon. It is expected that they will be available for general consideration after being revised in accordance with the discussions.

The Officers Committee held two meetings in December, 1952, and March, 1953, and issued a "D-13 News Letter" in January to inform members about progress and new developments between meetings. Further issues may appear semi-annually or as needed.

The following changes in subcommittee officers were approved by the Advisory Committee during the year: J. K. Frederick, Jr., vice A. M. Tenney, as chairman of Subcommittee A-2; J. H. Baldt as secretary of A-2, Section I; resignation of G. H. Hotte and W. E. Scholer as chairman and secretary, respectively, of A-2, Section II; F. W. Noechel, vice H. R. Anderson, as chairman of A-3, Section III; F. J. O'Neil as secretary of A-3, Section V; E. M. Simon, vice C. O. Werner, as secretary of A-9; G. B. Harvey, vice R. R. Stevens, as chairman of A-10; R. H. Carter, vice R. R. Stevens, as chairman of A-10, Section I with F. T. Campagna as secretary; T. F. Jacoby, vice E. A. Leonard, as chairman of A-11 with C. C. Fagan, vice F. T. Davies, as secretary.

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[*] Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Abstracts of papers by E. R. Frederick on "Corrugated Fibrous Webs as Insulation Fillers" and Herman Bogaty, Norman R. S. Hollies, John C. Hintermaier, and Milton Harris on "Thickness-Pressure Measurements and the Nature of a Fabric Surface" were published in ASTM BULLETIN, No. 188, February, 1953, pp. 45 and 46 (*TP 37* and *TP 38*).

The Joint ASTM-AATCC Committee on Textile Test Methods held two meetings in November, 1952, and March, 1953. In response to a request from Sectional Committee L14, the Joint Committee reviewed a number of test methods for rayon and acetate fabrics appearing in an American Standard developed by Sectional Committee L22 and which had not been approved as American Standard. The Joint Committee assigned certain of these methods to the AATCC and others to the ASTM, and in two instances to both organizations for review and study. The membership of the Joint Committee was increased by the addition of C. W. Dorn, representing AATCC, and W. M. Scott, representing ASTM.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative General Methods of:

Sampling Cotton Fibers for Testing (D 1441 - 52 T).

Tentative Method of:

Test for Fiber Weight per Unit Length and Maturity of Cotton Fibers (Array Method) (D 1442 - 52 T).

Tentative Recommended Practice for:

Designation of Yarn Construction (D 1244 - 52 T).

Revision of Tentative General Methods of:

Testing Cotton Fibers (D 414 - 49 T).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the new and revised tentatives appear in the 1952 Book of ASTM Standards, Part 7.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication 4 new tentatives and is recommending the revision of 6 tentatives, tentative revision of 1 standard, adoption as standard of several definitions, 3 tentatives, and tentative revisions of 2 standards, immediate revision of 2 standards, withdrawal of 1 standard, and withdrawal of 3 tentatives with one to be published as information only.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[2]

All other tentatives not specifically referred to are being actively studied by the respective sponsoring subcommittees.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-1 on Cotton and Its Products (B. L. Whittier, chairman):

Section I on Cotton (G. S. Buck, Jr., chairman) is continuing its study of the methods of test for various properties of cotton with a view to setting up separate standards for each property.

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) is studying methods of twist determination and preconditioning of yarns. Inspection of appearance standards is being continued and instructions regarding use of the photographic reference standards have been prepared.

Subcommittee A-2 on Man-Made Organic-Base Fibers and Their Products (J. K. Frederick, Jr., chairman):

Section I on Spun and Continuous Filament Yarns (J. K. Frederick, Jr., chairman) is studying regain bases for testing, revision of some of its standards, and the development of appearance standards.

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[2] See p. 445.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section II on Fabrics is undergoing reorganization and will study need for revision of standards under its jurisdiction.

Subcommittee A-3 on Wool and Its Products (Werner von Bergen, chairman):

Section I on Wool (O. P. Beckwith, chairman) is continuing its study of wool testing with particular reference to the determination of tensile strength, moisture content, staple length, alkali solubility, and fineness.

Section III on Woolen and Worsted Yarns (F. W. Noechel, chairman) is proposing a new tentative which is a consolidation of Methods D 403 and D 404 on woolen and worsted yarns and mixed yarns containing wool.

Section V on Woolen and Worsted Fabrics (E. M. Lynch, Jr., chairman) is studying reference regains for various fibers and the fiber analysis of blends.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman) is continuing its study of the significance of tests of asbestos materials and the analysis of asbestos textiles with particular reference to cotton and fiberglas.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (S. J. Hayes, chairman) is studying knot breaking strength and a general method of acceptance testing.

Subcommittee A-7 on Ultimate Consumer Textile Products (A. F. Tesi, chairman) is continuing its studies of test methods for pilling, elastic fabrics, and slide fasteners.

Subcommittee A-8 on Glass Fiber and Its Products (F. A. Mennerich, chairman) is continuing its study of methods for determining build-up and spread of glass yarns for wrap and braided covers of wires, and knot strength of glass yarns.

Subcommittee A-9 on Tire Cord and Fabrics (F. C. Kennedy, chairman) is continuing its study of methods of test for tensile strength of tire cords with particular reference to rayon cord.

Subcommittee A-10 on Non-Woven Fabrics (G. B. Harvey, chairman):

Section I on Felt (R. H. Carter, chairman) is continuing its study of stiffness of felts, and considering mildew resistance, wear abrasion, heat stability, and synthetic fibers in felt.

Section II on Bonded Fabrics (G. B. Harvey, chairman) is continuing its study on fire resistance, water absorbency, dry cleaning, abrasion resistance, tearing resistance, and flexural rigidity. Also, folding and scuff resistance, smoothness measurement, and resistance to delamination are being considered.

Subcommittee A-11 on Pile Fabrics (T. F. Jacoby, chairman) is working on service properties, fire resistance, resistance to insect damage, tests for tufted pile floor coverings, correlation of alkali solubility with damage to wool, moisture content of kraft cord, and tuft bind test.

Subcommittee B-1 on Test Methods (W. M. Scott, chairman) is continuing its study of dimensions of jaws for grab test, fiber properties, evenness of yarns, knit fabrics, crease resistance, thickness of fabrics, stiffness, fraying, identification of fibers, analysis of fiber mixtures, abrasion resistance, thermal transmission, and colorfastness.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman) is continuing its work of developing standard and tentative definitions, revision of the Glossary of Defects in Woven Fabrics. It will also continue its study of the designation of yarn construction and the universal yarn numbering system.

Subcommittee B-5 on Sampling, Presentation and Interpretation of Data (J. C. Hintermaier, chairman) is continuing its study of the sampling of part wool blankets for determining fiber content. The program for the October, 1953, papers session will be developed and sponsored by this subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 351 members; 182 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. D. APPEL,
Chairman.

W. H. WHITCOMB,
Secretary

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Tensile Strength of Wool Fiber Bundles (D 1294 – 53 T),
Test for Recovery of Textile Fabrics From Creasing, Using the Vertical Strip Apparatus (D 1295 – 53 T),
Test for Maturity of Cotton Fibers (Random Sample-Sodium Hydroxide Swelling Method) (D 1443 – 53 T),
Test for Cross-Sectional Characteristics of Cotton Fibers (D 1444 – 53 T),
Test for Strength of Cotton Fibers (Flat Bundle Method) (D 1445 – 53 T), and
Test for Number of Neps in Cotton Fibers (D 1446 – 53 T).

Revision of Tentative Methods of:

Testing Cotton Fibers (D 414 – 52 T),
Test for Resistance of Textile Materials to Microorganisms (D 684 – 45 T), and
Testing and Tolerances for Rayon Tire Cord (D 885 – 53 T).

These recommendations were accepted by the Standards Committee on October 30, 1953, and the new and revised tentative methods appear in the 1953 Supplement to Book of ASTM Standards, Part 7, as well as in the 1953 Compilation of ASTM Standards on Textile Materials.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1952 Book of ASTM Standards, Part 7.

NEW TENTATIVES

Committee D-13 recommends the following four methods for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:

Average Fiber Diameter of Wool Tops by Porous Plug Tester
Alkali Solubility of Wool
Relaxation and Felting Shrinkage in Laundering of Stabilized Knit Wool Fabrics
Testing and Tolerances for Yarns Containing Wool

REVISION OF TENTATIVES

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 52 T): On the recommendation of Subcommittee B-2, the committee recommends that these tentative definitions be revised by inclusion of the following additional items:

Break Factor, n.—A measure of yarn strength calculated as (*1*) The product of breaking strength times indirect yarn number, or (*2*) the product of breaking strength times the reciprocal of the direct yarn number.

NOTE 1.—Break factors are frequently based on skein strength expressed in pounds in the U.S.A.

NOTE 2.—Break factors for single strands measured in direct system yarn numbers, are directly related to Tenacity and to Breaking Length.

NOTE 3.—Break Factor is frequently given other designations as Strength Count Product, Lea Count Constant, Lea Product, Breaking Ratio, etc.

Fiber, n: 1. *General*—A generic term for the various types of matter (natural or man-made) which form the basic elements of textile fabrics and other textile structures.

NOTE.—Examples include cotton (fiber), glass (fibers or filaments), rayon (fibers or filaments), silk (filaments), wool (fibers), etc.

Filament.—Replace Note 1 following the definition for Filament with the following:

NOTE 1.—The extreme length of filaments permits their use as yarns with little or no additional twist and without the spinning operations required to convert fibers to yarns.

Hank, n: 1. A synonym for skein.

2. A definite length of slubbing, roving, or yarn. (The length depends on the yarn numbering system in use, for example, cotton hanks have a length of 840 yd.)
3. A term applied to slubbing or roving which is equivalent to the indirect yarn number (count), for example, a 1.5 hank roving.

Lea, n: 2. A unit of length used to determine the linear density of various spun yarns, usually a predetermined fraction of a hank for a specific yarn number system; for example, for cotton—120 yd.

Lea Skein, n: A skein having a length of 1 lea.

NOTE.—Lea skeins of a prescribed length and number of turns are used to determine the linear density and also the strength of yarns.

Length, Breaking, n.—After the definition currently printed in ASTM Designation D 123-52, add the following: "In Europe, breaking length is usually expressed in kilometers."

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[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

Misdraw, n: Fabric Defect.—A warp end that is not properly interlaced with the filling.

NOTE.—*Cause:* A warp end drawn through the wrong heddle when drawing in the warp, or when repairing a broken end following a break during weaving.

Reed Mark, n, Fabric Defect.—A mark running warpwise in the cloth due to crowded dents, open dents (cracks), damaged reed or improper reed.

Shiner, n, Fabric Defect.—A streak caused by a lustrous filament yarn.

NOTE.—Cause: The application of excessive tension to a yarn prior to, or during, its use in the loom.

Staple, adj. and *n.*—Natural fibers or cut lengths from filaments.

NOTE 1.—The staple length of natural fibers varies from less than 1 in. as in some cotton fibers, to several feet for some hard fibers.

NOTE 2.—The term "Staple (Fiber)" is used in the textile industry to distinguish natural or cut length (fibers) from filaments.

Sticker (Also—Hitch-back, Draw-back, Tie-back, or Warp Holding Place), *Fabric Defect, n.*—A distortion in the weave characterized by slack and tight places in the same warp yarns.

NOTE.—Cause: Warp ends restricted by broken filament slubs or knots catching at lease rod, drop wires, heddles or reeds.

Top, n: 1. *Worsted Process,* A sliver in which the fibers have been parallelized, and usually combed.

2. *Man-Made Fibers* or *Tow to Top Process.*—A sliver obtained by drafting or twisting, along with breaking or cutting a multifilament tow.

Tow, n: Man-Made Fibers.—A multifilament strand without twist and suitable for cutting into short lengths for use as staple fibers or as flock, or converting directly into a spun yarn.

Yarn Unevenness:

Linear Unevenness (*LUE*), *n.*—The non-uniformity of the linear density of a continuous strand or portion of a strand.

NOTE.—An estimate of the non-uniformity of linear density is usually made from measurements of some other property of the strand, such as variations in compressed cross-sectional area, variations in profile (usually measured photoelectrically), or changes in capacity of a condenser of which the fiber forms part of the matter between the plates.

Mean Linear Unevenness, ($\overline{LUE}$), *n.*—The average of the absolute values of the deviations of observed linear densities from the average linear density of a specific sample.

Mean Linear Unevenness, per cent, ($\overline{LUE\%}$), *n.*—The mean linear unevenness calculated as a percentage of the average linear density of a specific sample.

NOTE 1.—Mean Linear Unevenness, per cent, is usually measured by integrating meters or calculated from planimetered areas of an unevenness chart or an equivalent method.

NOTE 2.—Current expressions having an equivalent meaning are: Per Cent Mean Deviation and Average Variation.

Mean Range Unevenness, per cent, ($\overline{RUE\%}$), *n.*—The average of the ranges of linear density found in consecutive units of equal length of a strand, calculated as a percentage of the average linear density.

NOTE 1.—The term "Mean Range Unevenness, per cent" should be used for the result of the so-called "Hi-Lo" chart method in which $\overline{RUE\%}$ is calculated from the average of maxima and average of minima of consecutive units of equal length and the average linear density.

NOTE 2.—The value obtained in this method of evaluation depends upon the length of the consecutive units used. The $\overline{RUE\%}$ will increase as this length increases up to a point beyond which increased length has a negligible effect. When results are quoted, the length within which the range is measured should be noted along with the figure for $\overline{RUE\%}$.

NOTE 3.—Current expressions having an equivalent meaning are: Per Cent Unevenness and Per Cent Non-Uniformity.

Standard Deviation Linear Unevenness, (*SLUE*), *n.*—A quantitative measure of dispersion of linear density within a sample; the square root of the average of the squares of the deviations of linear density from the average linear density.

NOTE.—Standard Deviation Linear Unevenness is usually calculated from readings obtained from integrating meters which give the mean square deviation of the linear density from the average linear density of the sample.

Coefficient of Variation of Linear Unevenness, (*VLUE*), *n.*—A relative measure of dispersion of linear density within a sample; the standard deviation of the linear density expressed as a percentage of the average linear density.

Warp, n: 1. The yarn running lengthwise in a woven fabric.

2. A group of yarns in long lengths and approximately parallel, put on beams or warp reels for further textile processing including weaving, knitting, twisting, dyeing, etc.

Warp, Ball, n.—A warp put up in the form of a ball instead of on a beam.

Tentative Specifications and Methods of Test for Fineness of Wool Tops (D 472 – 50 T):

The committee recommends that this tentative be revised as follows:

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—PROPOSED REQUIREMENTS FOR WOOL TOPS.[a]

Grade	80's	70's	64's	62's	60's	58's	56's	54's	50's	48's	46's	44's	40's	36's
Fineness range, microns:														
Average diameter, min.	18.1	19.6	21.1	22.6	24.1	25.6	27.1	28.6	30.1	31.8	33.5	35.2	37.1	39.0
Average diameter, max.	19.5	21.0	22.5	24.0	25.5	27.0	28.5	30.0	31.7	33.4	35.1	37.0	38.9	41.2
Fibers, per cent:														
10 to 25 microns, incl, min.	91	83	....	....	....	....	....	....	....	....	....	....	....	....
10 to 30 microns, incl, min.	....	....	92	86	80	72	62	54	44	....	....	....	....	....
10 to 40 microns, incl, min.	....	....	....	....	....	....	....	....	....	75	68	62	54	44
25.1 and over, microns, max.	9	17	....	....	....	....	....	....	....	....	....	....	....	....
30.1 and over, microns, max.	1	3	8	14	20	28	38	46	56	....	....	....	....	....
40.1 and over, microns, max.	....	....	1	1	2	....	....	....	....	25	32	38	46	56
50.1 and over, microns, max.	....	....	....	....	....	1	1	2	2	....	....	....	....	....
60.1 and over, microns, max.	....	....	....	....	....	....	....	....	....	1	1	2	3	4
Minimum number of fibers required for test, per sample	400	400	600	600	800	800	1000	1000	1000	1600	1600	1600	1600	1600

[a] The minor percentage group is a part of, and not in addition to, the first maximum group. The combined groups should not exceed 100 per cent.

Tentative Methods of Testing and Tolerances for Rayon Tire Cord (D 885 – 46 T):

The committee recommends that these methods be revised as appended hereto.[2]

Tentative Methods of Test for Clean Wool Content of Wool in the Grease (D 584 – 52 T):

The committee recommends that this tentative be revised as follows:

Title.—Change to read as follows: "Tentative Methods of Test for Total Wool Fiber Present In Wool In The Grease."

Section 1.—Insert "fiber" before "contained."

Section 2.—Change the beginning of this Section to read: 2. *Wool Fiber Content.*—The total wool fiber content of a sample shall be. . . ."

Section 5.—Add as a new Note 4 the following:

NOTE 4.—If "clean content" (see Note following Section 17) also is to be calculated, loss of vegetable matter at any stage of the test must be avoided.

Section 6.—Change "Hard Scoured Wool Content" to "Total Wool Fiber Present."

Section 9.—In the last sentence of Paragraph (*b*) omit "residual." Change the beginning of Paragraph (*f*) to read: "(*f*) *Vegetable Matter.*—Determine the percentage of oven-dry, ash-and-extractives-free vegetable matter in the. . . ."

Section 10.—Throughout this section, change "clean wool content" to "wool fiber content." (There are seven such changes.)

In the definitions of the symbols

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[2] The revised methods were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

"V_T" and "V_1, V_2, V_3" insert "oven-dry, ash-and-extractives-free" before "vegetable matter."

Sections 11, 16, and 17.—Change "clean wool content" to "wool fiber content." (There are seven such changes.)

Note.—Add the following new Note:

NOTE.—For import-duty purposes, the term "clean content" of wool as defined by the Bureau of Customs means the *yield* of clean wool, adjusted to standard conditions (Section 3), that is obtainable from imported wool or hair when cleansed by usual commercial methods. The term does not include the weight of any wool fibers unavoidably lost or rendered unusable in the course of ordinary commercial cleansing operations.

To obtain the percentage of clean content from the percentage of total wool fiber present as determined by this method (or an equivalent method), the Bureau of Customs deducts an allowance for estimated average wool fiber loss, calculated as follows:

$$L = 0.005\,C + 0.6\,VM \qquad (5)$$

where:

L = allowance for wool fiber loss, as a percentage of the greasy weight, to be deducted from C,

C = percentage of wool fiber content (from Eq. 4), and

VM = percentage of vegetable matter, adjusted to standard condition (Section 3), present in the wool in the grease.

For the calculation of VM from V_1, V_2, V_3, V_T (Section 10), Eq. 6 may be used, as follows:

$$VM = \frac{W_B(P_1V_1 + P_2V_2 + P_3V_3 + \cdots)}{0.86W(W_1 + W_2 + W_3 + \cdots)} + \frac{P_TV_T}{0.86W} \cdots \qquad (6)$$

Tentative Methods of Core Sampling of Wool in Packages for Determination of Percentage of Hard Scoured Wool Content (D 1060 – 49 T):

The committee recommends that this tentative be revised as follows:

Title.—Change "Hard Scoured Wool Content" to "Total Wool Fiber Present."

Section 1.—Change the first sentence to read as follows: "These methods of sampling are intended for use in obtaining a sample of a lot of wool in bales or bags for the determination of the percentage of wool fiber content by the Tentative Methods of Test for Total Wool Fiber Present in Wool in the Grease (ASTM Designation: D 584),[3] or an equivalent method."

Sections 2, 3, 4, 5.—Wherever the expression "hard scoured wool content" appears, change to "wool fiber content."

Tentative Recommended Practice for Designation of Yarn Construction (D 1244 – 52 T):

The committee recommends the following revisions in this recommended practice:

Section 3.—Number the examples illustrating the symbols used to designate structure in Section 3, and now printed after Sections 3(*a*), (*b*), (*c*), (*d*) and (*e*), and print all of them at the end of Section 3, and add appropriate references at the end of the individual paragraphs in Section 3.

After Paragraph (*e*) add the following examples:

Examples:

1. Cotton Count (Br.) 24:Z15 tpi
2. Cotton Count (Br.) 24:Z15 / 2:S8 tpi
3. Worsted Count 50:Z17 / 2:S11 tpi
4. Grex Units 325:Z12 x 2:S13.5 tpi
5. Cotton Count (Br.) 23:S20 / 5:Z18 / 3:S14 tpi
6. Worsted Units 50/2
7. Cotton Count (Br.) 23/5/3
8. Cotton Count (Br.) 24:Z17 / 5:S13 tpi
9. Denier Units 150:S3 x 3:Z7 tpi
10. Cotton Count (Br.) 24:Z17 / 5:S17 tpi

This revision is proposed because it is not possible to illustrate only a single rule in an example and some of the first examples included the use of symbols covered by rules which are given in later paragraphs.

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[3] 1952 Book of ASTM Standards, Part 7.

Section 4(a).—Add the following new Paragraph (*3*), renumbering the remaining ones accordingly:

(*3*) Where the single yarn designated is a spun yarn made from man-made fibers, the average linear density and length of the fibers shall be indicated. When these properties are known for natural fibers, they shall be indicated in a similar manner.

Examples:

1. Cotton Count (Br.) 24:Z15 Cotton 15/16″ staple
2. Cotton Count (Br.) 30:Z18 tpi Bright Rayon 1.5 d/f, 1.5″ long
3. Grex 200:S 16 tpi Acetate 3.3 d/f, 3″ long

Revise Paragraph (*3*) renumbered as (*4*) to read as follows:

(*4*) If the single yarn has been spun from two or more fiber types (a blended yarn) the nominal percentage of the different fibers shall be noted after the conventional designation, and enclosed in parentheses. When known, the average linear density and length of the fibers shall be noted also.

Example: Worsted 45:S17 (wool 80%, 58's and dull acetate count 20%, 1.5 d/f, 3″ long)

The above revised Sections 4(*a*) (*3*) and (*4*) are proposed to cover details of the length and average linear density of the fibers used in the manufacture of the spun yarns.

TENTATIVE REVISION OF STANDARD

Standard General Methods of Testing Woven Fabrics (D 39 - 49):

The committee recommends the following tentative revision of this standard:

Section 10(b).—Change the third sentence to read as follows:

The face of one jaw of each clamp measure 1 by 1 in. or 1 by 2 in., the longer dimension being parallel to the direction of the application of the load. The other jaw of each clamp shall be at least the same dimensions as stated above. Each set of jaws of each clamp, and the two clamps, shall be in line.

ADOPTION OF TENTATIVES AS STANDARDS

Tentative Definitions of Terms Relating to Textile Materials (D 123 - 52 T):

On the recommendation of Subcommittee B-2, the committee proposes that the definitions for the following 21 items now appearing in Tentative Definitions D 123 - 52 T be approved for submission to letter ballot of the Society for adoption as standard:

1. Crimp,
2. Fiber Number,
3. Filament,
4. Filament Number,
5. Fineness,
6. Grex Unit,
7. Load, Breaking,
8. Moisture,
9. Pick Count,
10. Pre-tension,
11. Skein, Numbering,
12. Tensile Test,
13. Tex Unit,
14. Weight, Commercial,
15. Weight, Moisture-free,
16. Wool,
17. Yarn, Blended,
18. Yarn-Numbering Systems—Grex, Metric, and Tex, and
19. Yarn, Single.

The following editorial changes are recommended in two of the items listed above: In Item 15, Weight, Moisture-free, in the last line insert a comma after the word "lot." In the definition for Yarn, Blended, change the cross-reference to read (See also Yarn, Combination and Yarn, Self-blended). In the example following Yarn, Blended, change the word "synthetic" to "man-made."

It is also recommended that the definitions for the following items currently printed in Tentative Definitions D 123 - 52 T be printed in the Glossary given as Appendix I of Standard D 123:

1. Flock,
2. Fly,
3. Merino,
4. Twist, Set, and
5. Twist, Zero.

Tentative Methods of Test for Apparent Fluidity of Dispersions of Cellulose Fibers (D 539 – 51 T),

Tentative Methods of Testing Bonded Fabrics (D 1117 – 50 T), and

Tentative Methods of Testing Twine (From Bast and Leaf Fibers) (D 1233 – 52 T):

The committee recommends that these three tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Definition of Terms Relating to Textile Materials (D 123 – 52):

The committee recommends for immediate adoption changes in the definitions for "Barré" and "Crack" which currently appear in Appendix II on Defects in Woven Fabrics, of Standard D 123 – 52, and the following editorial changes in the definitions for Filling, Pulled-in and Slug:

Barré, n.—A striped effect in a fabric caused by a series of one or more picks which are characterized by an apparent difference in color or luster that is repeated at intervals in the warp direction. The effect may be a real difference in color or luster, or only an apparent difference due to fabric geometry.

Crack (Also Open Dent), *n.*—An open streak of variable length parallel with the filling or with the warp.

Filling, Pulled-in.—Add the following alternatives after the current title: (Also; Filling, Jerked-in; Filling, Whipped-in; and Filling, Dragged-in.)

Slug, after the title insert the following alternative: (Also Slub).

Delete from Appendix II the definition, causes and picture for Item 4, *Cracked* or *Open Dent.* Number item 5 as item 4 and change all subsequent numbers accordingly.

Standard Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 – 52):

The committee recommends the following revision of this standard for immediate adoption and accordingly asks for the necessary nine-tenths favorable vote at the Annual Meeting for submission of this recommendation to letter ballot of the Society:

RELAXATION AND FELTING SHRINKAGE TEST

16.(*a*) *Scope.*—This method of test is designed for testing the relaxation and felting shrinkage of woven wool and part wool fabrics.

(*b*) *Principle.*—Marked specimens are sprayed with water, extracted, dried by pressing, and measured to determine relaxation shrinkage. The same specimens are then washed in a machine under controlled conditions, extracted, dried by pressing, and again measured to determine total shrinkage. Total shrinkage less relaxation shrinkage equals felting shrinkage.

(*c*) *Apparatus and materials:*

(*1*) *Cylindrical Washing Machine,* of the reversing type.[4] The wheel or cage shall be 20 to 24 in. in inside diameter, and 20 to 24 in. in inside length. There shall be three fins, each approximately 3 in. wide, extending the full length of the inside of the wheel. One fin shall be located every 120 deg around the inside diameter of the wheel. The wash wheel shall rotate at a speed of 30 rpm, making 5 to 10 revolutions before reversing, and shall be equipped with a pipe for injecting live steam to raise the temperature of the water in the machine. The machine shall contain an opening for the insertion of a thermometer or other equivalent equipment for determining the temperature of the water during the washing and rinsing procedures. It shall also be equipped with an outside water gage that will indicate the level of the water in the wheel.

(*2*) *Flat-Bed or Steam Press,*[5] having a steam pressure of 60 psi.

(*3*) *Soap,* conforming to the Standard Specifications for Chip Soap (ASTM Designation: D 496). Stock solution, containing 1 lb of chip soap to 1 gal of water, may be used.

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[4] Washing machines of this type may be obtained from the American Laundry Machine Co., Cincinnati, Ohio; Robert Ewing & Sons Co., Green Island, N. Y.; and the Prosperity Co., Syracuse, N. Y.

[5] Suitable pressing equipment is obtainable from the General Electric Co., Schenectady, N. Y.; Young & Franklin Tool Works, 107 N. Franklin St., Syracuse, N. Y.; the Prosperity Co., Syracuse, N. Y.; and the Pantex Co., Pawtucket, R. I.

(*4*) *Marking Device and Graduated Scale*,[6] for measuring shrinkage.

(*d*) *Test Specimens.*—The specimens of cloth taken for test shall be at least 20 in. square. The specimens shall be fully conditioned in a standard atmosphere having a relative humidity of 65 ± 2 per cent at 70 ± 2 F (21 C), as prescribed in Method D 39. They shall be laid out without tension on a flat, hard surface, care being taken that the cloth is smooth and free from wrinkles or creases. Three distances, each of 18 in., shall be marked off on the specimens in both the warp and filling directions by the marking device. Satisfactory marks may also be made with indelible ink and a fine-pointed pen, or by sewing fine threads into the cloth. The distances shall be parallel to the warp or filling threads, at least 6 in. apart, and at least 1 in. from all edges of the specimen.

(*e*) *Procedure for Determination of Relaxation Shrinkage.*—The test specimen, after marking, shall be placed in a sink or similar apparatus of such shape and size as to allow the specimen to be laid flat without folding. Then the specimen shall be thoroughly saturated by spraying with water at a temperature of 75 to 85 F (24 to 29 C), using an ordinary garden hose and nozzle with the nozzle adjusted to a spray position. It is essential in the spraying operation to prevent undue distortion of the specimen, such as would result from picking up the wet fabric by one corner. Therefore, the best method of handling the wet fabric is by turning in the edges and corners of the specimen, and picking it up as a ball. After spraying, the specimens shall be hydroextracted with as little distortion as possible. After extracting, each specimen shall be opened on the buck of the press without any stretching, and pressed dry. The pressed specimens shall be laid out without tension on a flat, hard surface, care being taken that the cloth is smooth and free from wrinkles or creases, and allowed to fully recondition at standard atmospheric conditions. The distances which were marked off (see Paragraph (*d*)) shall then be measured again. The shrinkage in the warp and in the filling directions of the cloth shall be calculated separately as the average obtained from the three marked distances. The change in dimension shall be measured with the steel scale, graduated directly in percentage, and the result shall be recorded as relaxation shrinkage.

(*f*) *Procedure for Determination of Felting Shrinkage.*—The same specimens, and such additional cloth as may be necessary to make a 3-lb load, shall be placed in the wash wheel. The required amount of water at 100 F (38 C) and of soap shall then be added for the sudsing operation. A 7 in. water level shall be used for both sudsing and rinsing operations, and the amount of soap used shall be sufficient to produce a head of suds at least 1 in. high. In general, the average strength of the soap solution shall be about 0.1 per cent. The wash wheel shall be kept running continuously for 75 min. from the start of the test, and the temperature of the wash liquor shall be maintained at 100 F during the 75-min sudsing operation. The washer shall then be stopped and drained. The wheel shall be filled with the necessary amount of clear rinsing water at 100 F and run for 5 min. The machine shall be stopped again, drained, and the rinsing operation repeated. After the final rinse the wash wheel shall be drained, and the specimens transferred to the centrifuge, each one handled as a ball. The specimens shall then be extracted and pressed, as described under the procedure for relaxation shrinkage (Paragraph (*e*)). The pressed specimens shall be laid out without tension on a flat, hard surface, care being taken that the cloth is smooth and free from wrinkles or creases, and allowed to fully recondition at standard atmospheric conditions. The distances which were marked off shall be measured again as described in Paragraph (*e*), in order to determine total shrinkage. Total shrinkage less relaxation shrinkage equals felting shrinkage.

ADOPTION AS STANDARDS OF TENTATIVE REVISION OF STANDARDS

The committee recommends that the tentative revision[3] of Sections 8 (*e*) and 14 (*a*) of the Standard Methods of Testing Felt (**D 461 – 51**) be approved for submission to letter ballot of the Society for adoption.

The committee recommends that the tentative revision[3] of the Standard Specifications for Textile Testing Machines (**D 76 – 49**) which covers proposed changes in Section 2 on Classes of Machines, in Section 3 on Machine Operation, and a new Section 8 on Machine Operation for Constant-Rate-of-Specimen-Extension Tests be approved for reference to letter ballot of the Society for adoption as standard.

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[6] A mechanical marking device and graduated scale may be purchased through the Sanforizing Div., Cluett-Peabody & Co., Inc., Troy, N. Y.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications and Methods of Test for Osnaburg Cement Sacks (**D 205 - 39**).

WITHDRAWAL OF TENTATIVES

The committee recommends that the Tentative Methods of Test for Identification of Finishes on Textiles (**D 683 - 42 T**) be discontinued as tentative and reverted to the status of a proposed method and published as information.

The committee also recommends withdrawal of the Tentative Specifications and Methods of Test for Woolen Yarns (**D 403 - 48 T**) and for Worsted Yarns (**D 404 - 48 T**) which are being replaced by the Tentative Methods of Testing and Tolerances for Yarns Containing Wool.[7]

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[7] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

REPORT OF COMMITTEE D-14
ON
ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year, one at ASTM Headquarters in Philadelphia, Pa., on October 30 and 31, 1952, and one in Cincinnati, Ohio, on April 9 and 10, 1953.

At the present time, Committee D-14 consists of 96 members, of whom 35 are classified as producers, 39 as consumers, and 22 as general interest members. There are also 3 consulting members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-14 presented the revision of Tentative Method of Test for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (D 1002 – 49 T) to the Society through the Administrative Committee on Standards. This revision was accepted by the Standards Committee on March 17, 1953.[1]

On May 20, 1953, the Administrative Committee on Standards accepted the following definitions as a tentative revision of the Standard Definitions of Terms Relating to adhesives (D 907 – 52):

Plasticity.—In the case of adhesives, a property which allows the material to be deformed continuously and permanently without rupture upon the application of a force that exceeds the yield value of the material.

Primer.—In the case of adhesives, a coating applied to a surface, prior to the application of the adhesive, to improve the performance of the bond.

NEW TENTATIVE

Committee D-14 recommends the publication of a new Tentative Method of Test for Effect of Mold Contamination on Permanence of Adhesive Preparations and Adhesive Bonds, as appended hereto.[2]

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Blocking Point of Potentially Adhesive Layers (**D 1146 – 51 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status without revision, pending further evaluation work and study:

Recommended Practice for:

Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (**D 904 – 46 T**),

Determining Strength Development of Adhesive Bonds (**D 1144 – 51 T**), and

Determining the Effect of Moisture and Temperature on Adhesive Bonds (**D 1151 – 51 T**).

Method of Test For:

Consistency of Adhesive (**D 1084 – 50 T**),

Effect of Bacterial Contamination on Permanence of Adhesive Preparations and Adhesive Bonds (**D 1174 – 51 T**),

Resistance of Adhesives for Wood to Cyclic Accelerated Service Conditions (**D 1183 – 51 T**), and

Strength to Adhesives on Flexual Loading (**D 1184 – 51 T**).

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The revised method appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

[2] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

[3] 1952 Book of ASTM Standards, Part 7.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (C. B. Hemming, chairman) has been greatly interested in a method of butt-joint tension testing presented under the Section on Tensile Strength. Not only is the method suited for round or square specimens, but it can be modified for other types of test. Extensive revision of Method D 1184 - 51 T is under way by the Section on Flexural Strength.

Subcommittee II on Analytical Tests (C. K. M. Winne, chairman) is trying to get sufficient data together to determine the best modification of a method of determining the nonvolatile content of urea resin adhesives.

Subcommittee III on Permanence (R. F. Blomquist, chairman).—The Section on Cyclic Tests is working on revisions of Method D 1183 - 51 T. The subcommittee recommended that Recommended Practices D 1151 - 51 T and D 904 - 46 T be retained as tentatives, since it was believed that neither of these has as yet been extensively used. The method on mold effect, appended hereto,[2] has been presented by the Section on Biological Factors.

A questionnaire has been mailed to the members of Committee D-14 by the Section on Correlation, Long and Short Term Tests, to enlist assistance.

Subcommittee IV on Working Qualities (M. Petronio, chairman).—The Section on Consistency and the Section on Storage and Working Life have each submitted two methods for letter ballot of Committee D-14. All four of these methods are currently being revised for resubmittal to the committee. The Section on Tack and Blocking has been instructed to submit a method on tack to the subcommittee.

Subcommittee V on Specifications (F. H. Bair, chairman).—The Section on Acoustical Materials asked that Committee D-14 arrange if possible a joint jurisdiction with Committee C-20 on this subject. A Section on Wood Adhesives plans to start work on adhesives for marine laminants, where appreciable interest exists. A Section on Sampling and Inspection has been established.

Subcommittee VI on Nomenclature (F. W. Reinhart, chairman).—Definitions of the two terms "plasticity" and "primer" have been approved by the subcommittee and are now before the Administrative Committee on Standards. The cooperation of Committee D-7 on Wood has been requested on the nomenclature relating jointly to wood and adhesives.

Subcommittee VII on Research (R. D. Platow, chairman).—Mr. Alan A. Marra of the University of Michigan discussed at the 1952 Fall Meeting some aspects of his work in the study of wood-to-wood gluing. This was of such interest that he was asked to continue on this general subject at the 1953 Spring Meeting.

Subcommittee VIII on Electrical Properties (H. A. Perry, chairman).—A proposed tentative method of test for adhesives relative to their use as electrical insulation has been recommended for letter ballot of Committee D-14.

This report has been submitted to letter ballot vote of the committee, which consists of 96 voting members; 74 members have returned their ballots, of whom 60 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

GERALD REINSMITH,
Chairman.

L. M. PERRY,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-15
ON
ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held two meetings during the year: the first in New York, N. Y., on October 17, 1952, and the second in Washington, D. C., on April 8, 1953. The Advisory Committee and subcommittees have held meetings concurrently.

There have been four resignations and four additions to the list of committee members during the year. At present the committee is composed of 41 active voting members, of whom 20 are classified as producers, 9 as consumers, and 12 as general interest members.

The following changes in subcommittee chairmanships occurred during the year: N. W. Faust vice J. D. Klinger as chairman of Subcommittee II on Antifreeze Field Testers; O. W. Chandler vice P. I. Emch as chairman of Subcommittee V on Effect of Antifreeze on Rubber Hose, also A. E. Williams replaced E. F. Miller as the liaison member on this Subcommittee V with Subsection III-B on Coolant System Hose of the SAE-ASTM Technical Committee on Automotive Rubber of Committee D-11; Gus Kaufman vice R. E. Mallonee as chairman of Subcommittee VIII on Editorial and Coordination of Test Methods.

NEW TENTATIVE

Committee D-15 recommends that the Method of Test for pH of Concentrated Engine Antifreezes be accepted for publication as tentative, as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD

Committee D-15 recommends that the following two tentative methods be adopted as standard without revision:

Tentative Methods of Test for:

Boiling Point of Engine Antifreezes (D 1120 – 51 T),[2] and

Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (D 1122 – 50 T).[2]

REVISION OF TENTATIVES

Tentative Specifications for Hydrometer-Thermometer Field Tester for Engine Antifreezes (**D 1124 – 50 T**).[2]—The committee recommends the following revision of these specifications:

Section 10(a). Revise the first sentence to read: "Determine the accuracy of the antifreeze tester freezing protection readings by comparison with solutions of the type or individual products, or both, of antifreeze or antifreezes of known freezing point for which the tester is designed."

Tentative Method of Test for Ash Content of Concentrated Engine Antifreezes (**D 1119 – 50 T**).[2]—The committee recommends the following revision of this method:

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 5.

[2] 1952 Book of ASTM Standards, Part 5.

Section 4. Add a note under Paragraph (*c*) to read as follows:

NOTE.—If desired, muffle furnace ignition may be used in place of the Meker burner. If a muffle furnace is used, proceed as specified in Section 4(*a*) and (*b*), and then complete the procedure as follows: When the liquid has been distilled or burned off, and the crucible appears dry, transfer the crucible to a muffle furnace (to prevent losses from spattering due to rapid burning or volatilization of traces of volatile constituents which may remain, the muffle furnace should be below 200 C when this transfer is made). Raise the temperature at the maximum rate to between 750 and 900 C and ignite for 1 hr. Cool the crucible, cover, and weigh to the nearest 0.1 mg. Then proceed as specified in Section 4(*d*).

TENTATIVES CONTINUED WITHOUT REVISION

The Committee recommends that the following four tentative methods be continued as tentative without revision:

Tentative Methods of:

Sampling and Preparing Aqueous Solutions of Engine Antifreeze for Testing Purposes (D 1176 - 51 T),
Test for Freezing Point of Aqueous Engine Antifreeze Solution (D 1177 - 51 T),
Test for Reserve Alkalinity of Concentrated Engine Antifreezes (D 1121 - 50 T), and
Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 - 50 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Freezing Point Determination (R. E. Mallonee, chairman) has continued consideration of thermometers for use with the freezing point method in cooperation with Subcommittee 17 on Thermometers of ASTM Committee E-1 on Methods of Testing.

Subcommittee II on Antifreeze Field Testers (J. D. Klinger, chairman) has recommended a minor change in Method D 1124 - 50 T to indicate plainly that the precision applies only to the product for which the tester is designed. In addition, consideration is being given to instructions or precautions in the use of the tester.

Subcommittee III on Physical Properties (R. E. Vogel, chairman) reviewed the experience with Methods D 1120 - 51 T, Test for Boiling Point of Engine Antifreezes, and Method D 1122 - 50 T, Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer, and found them satisfactory for adoption as standard. Methods for determining the solubility of engine antifreezes in water have been studied.

Subcommittee IV on Chemical Properties (E. G. Travis, chairman) completed work on, and recommended for publication as tentative, the procedure for the Determination of pH of Engine Antifreeze referred to previously. As a result of work done in the committee, a revision of the Method of Test for Ash Content of Concentrated Engine Antifreeze (D 1119 - 50 T) to permit the alternate use of a muffle furnace in the ashing step was recommended as indicated above. Preliminary screening work on procedures for the determination of total solids of engine antifreezes was carried out and a study group to further the investigation was formed. A project for the development of methods for determining individual glycols or glycol types present in glycol engine antifreeze was activated. A study group to deal with this problem was formed, but active work had not been started at the time of the preparation of this report.

Subcommittee V on Effects of Antifreeze on Rubber Hose (O. W. Chandler, chairman) is continuing in the attempt to develop a simple laboratory rubber immersion test for the evaluation of this property. The results of the previous two series of tests by the SAE-ASTM immersion test have been critically evaluated. Poor correlation with field service was indicated. A third series of tests on two modifications of the SAE-ASTM procedure is being undertaken. The modified tests are being run in an effort to obtain closer approach to field service conditions.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Simulated and Actual Service Testing (E. F. Harford, chairman).—The Study Group on Corrosion and Foaming Test Methods (C. H. Sweatt, chairman) has completed the series of cooperative shake-down tests on evaluation of several variables with the glassware corrosion test apparatus. The optimum conditions with respect to test duration, corrosion coupon spacing, temperature, aeration rate, and solution volume have been determined. Cooperative tests with these optimum conditions in about a dozen laboratories with five coolants have been planned.

This report has been submitted to letter ballot of the committee, which consists of 41 voting members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. R. WOLF,
Chairman.

C. F. GRAHAM,
Secretary.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held one meeting during the year: on June 26, 1953, at New York, N. Y. No separate subcommittee meetings were held, but the work of each subcommittee was reported by the chairman and fully discussed by all members during one general committee meeting.

Since the immediate objectives of Subcommittees III on Volatile Oil in Rosin and VI on Pine Tar and Tar Oil have been accomplished by the adoption of standard methods of test, these two subcommittees were placed in temporarily inactive status.

The committee has lost two members by resignation, namely, A. L. Brogden, Turpentine and Rosin Factors, Inc., representing the producers, and L. B. Parsons, Lever Bros. Co., who was a consumer representative but whose company no longer uses rosin.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-17 presented to the Society through the Administrative Committee on Standards proposed Tentative Methods of Test for Rosin Acids in Fatty Acids. The methods were accepted by the Standards Committee on September 23, 1952, and they appear in the 1952 Book of ASTM Standards, Part 4, bearing the designation D 1240-52 T.

ADOPTION OF TENTATIVE AS STANDARD

Committee D-17 recommends that the Tentative Methods of Testing Rosin Oils (**D 1131 - 50 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard, with addition of the following Note at the end of Section 9 on Volatile Matter:

NOTE.—Wide deviations in the percentage of volatile matter in rosin oil may result when different types of ovens are used for this test. If a purchaser of rosin oil establishes a limit for volatile matter, the type of oven used, whether convection or forced draft, should be clearly indicated.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Unsaponifiable Matter in Rosin (**D 1065 - 51 T**) be continued as tentative without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—Two members of the subcommittee have participated in the collaborative studies being made in Committees E-1 on Methods of Testing and D-4 on Road and Paving Materials, the purpose of which is to reach a basis for consolidation of the two ASTM Ring-and-Ball Softening Point Methods (D 36 - 26 and E 28 - 51 T), into a single ASTM standard. Progress is also reported in the development of a procedure for preparing a ring sample of synthetic resins by a "powder press" method, where preliminary melting to pour the ring mold is not possible.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A letter of inquiry to the members of the subcommittee regarding collaborative test work on softening point of rosin by means of a penetrometer test, as used in Method D 5 – 49, or by the Dennis-Parr electrically heated bar,[3] brought out the fact that such apparatus was not available to enough members of the committee to permit collaborative work at this time. It is hoped that data may eventually be obtained to show how the softening point of rosin by the Dennis bar method compares with the present standard ASTM ring-and-ball method.

Subcommittee IV on Chemical Analysis of Rosin (R. Herrlinger, chairman).—Several members of this subcommittee have made further tests of the proposed separatory funnel method for determining unsaponifiable matter in rosin.[4] A questionnaire developed the fact that seven members of the committee make occasional determinations of unsaponifiable matter in rosin and have agreed to participate in further work, it being felt that a separatory funnel method should be made available as an ASTM standard, even if it is not as fully suited for referee application as is the continuous extraction method. It was also felt by most of the collaborating members that the conditions under which the saponification of the rosin sample is carried out in the Methods of Test for Saponification Number of Rosin (D 464 – 51) should also be followed in working out any procedure for determining unsaponifiable matter by the funnel method, especially since half normal alkali is used to saponify the rosin in the present Tentative Method of Test for Unsaponifiable Matter in Rosin (D 1065 – 51 T).

Subcommittee V on Tall Oil (A. Pollak, chairman).—The details and description of the new method for rosin acids in refined tall oil (D 1240 – 52 T), referred to on the preceding page, applicable to low rosin-acid oils, was published in the *Journal* of the American Oil Chemists Society, August, 1952. The authors, who are members of the subcommittee, will now participate and supervise further collaborative work, which, if the results are satisfactory, can be the basis for incorporation of this new method into the present Standard Methods of Testing Tall Oil (D 803 – 51), for use on tall oils of low rosin-acid content. A member of the committee has reported appreciable variation in the value for rosin acids in tall oil, by the present Modified Wolff Method, if the procedure followed in cooling and titrating after esterification is varied. Clarification of the language of the test, as given in Methods D 803, is being considered. A study of methods for determining quantitatively the rosin content of varnishes is under way in a special task group of Subcommittee IX on Varnish, of Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

Subcommittee VIII on Rosin Oil (V. E. Grotlisch, chairman).—The subcommittee is recommending that the Tentative Methods of Testing Rosin Oils (D 1131 – 50 T) be adopted as standard. In view of criticism of the instructions given for the determination of volatile matter, it is proposed to include a note in Section 9 of the method, as indicated above.

Subcommittee X on Specifications (S. R. Snider, chairman).—The subcommittee has recommended that the Proposed Specifications for Rosin be published as tentative as printed in the 1952 Report of the Committee. Recommendation is also made that the Specifications for Dipentene be also published as tentative. Due to divergence of opinion on the scope and definition of pine oil, this material will require further study and discussion by the full committee before a recommendation can be made thereon.

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[3] Briefly described in *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 474 (1947).

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 391 (1950).

[NOTE.—The committee at the 1953 meeting at Atlantic City voted to withold approval of these recommendations at this time, pending a further study of the advantages and disadvantages to producers and consumers of having ASTM specifications for these naval stores products.]

This report has been submitted to letter ballot of the committee, which consists of 37 voting members; 27 members returned their ballots, of whom 25 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held its annual meeting at New York, N. Y., on June 27, 1952. An Executive Subcommittee meeting was held on January 11, 1953.

Two symposiums were sponsored at the 1952 Annual Meeting of the Society. The first, entitled "Direct Shear Testing of Soils," occupied one session and included seven papers. The second symposium on "Exchange Phenomena in Soils" occupied one session and consisted of five papers. All the papers and discussions are being published by the Society as special technical publications (*STP Nos. 131 and 142*).

The following publications were sponsored by the Committee and published during the past year by the Society:

Surface and Subsurface Reconnaissance (*STP No. 122*),
Consolidation Testing of Soils (*STP No. 126*),
Direct Shear Testing of Soils (*STP No. 131*),
Use of Radioisotopes in Soil Mechanics (*STP No. 134*),
Exchange Phenomena in Soils (*STP No. 142*), and
Bibliography on Soil Dynamics (*STP No. 146*).

Committee D-18 now consists of a total membership of 125 individual members and 17 consultants. There are 97 voting members, divided as follows: 17 producers, 43 consumers, and 37 general interest members.

The death of Mr. Lawrence Ortolani, member of the committee, was announced at the June meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-18, jointly with Committee D-4 on Road and Paving Materials, presented to the Society, through the Administrative Committee on Standards, proposed Tentative Specifications for Materials for Soil-Aggregate Sub-Base, Base, and Surface Courses. The specifications were accepted by the Standards Committee on September 5, 1952 and appear in the 1952 Book of ASTM Standards, Part 3, bearing the designation D 1241 - 52 T.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following six tentatives, which have stood for 2 years or more without revision, be continued in their present status without change:

Tentative Definition of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T).—Subcommittee G-3 is working on revisions.

Tentative Method of Testing of Soil-Bituminous Mixtures (D 915 - 47 T).—Study of this tentative method is desirable through the correlation of construction control and service performance records. The Joint Subcommittee of D-18 and D-4, which has jurisdiction over this tentative, will assemble data on the correlations.

Tentative Method of Test for Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burggraff Shear Apparatus (D 916 - 47 T).—Considerable data have been obtained on the use of this apparatus. It is expected that, when these data are correlated with the service behavior of the soils, a revision of the method may be indicated.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

Tentative Method of Test for Amount of Material in Soils Finer than the No. 200 Sieve (D 1140 – 50 T).—A letter ballot for the adoption as standard of this method will probably be recommended at the next meeting.

Tentative Method for Static Loading Test of Piles (D 1143 – 50 T).—The committee recommends that this method remain as a tentative until practical experience indicates any necessary changes.

Tentative Method of Test for Moisture Density Relations of Soils (D 698 – 42 T). Revisions of this tentative method are now in preparation by the originating subcommittee.

WITHDRAWAL OF TENTATIVES

In view of the publication of the Tentative Specifications for Materials for Soil-Aggregate Sub-Base, Base, and Surface Courses (D 1241 – 52 T), which was accepted on September 5, 1952, Committee D-18 recommends the withdrawal of the Tentative Specification for Materials for Stabilized Base Course (D 556 – 40 T) and Tentative Specification for Materials for Stabilized Surface Course (D 557 – 40 T).

SUBCOMMITTEE ACTIVITIES

Subcommittee G-3 on Nomenclature and Definitions (Charles F. Foster, chairman) held two meetings during the past year, in Denver on June 19, 1952, and in Vicksburg on March 9, 1953. The subcommittee has been following its original plan for each member to search the literature, record various definitions given by authorities, and recommend definitions and the symbols for the terms assigned to them. The work has been slow because the compilation of definitions for each term requires considerable research. The ASCE Committee on Definitions and Symbols compilation of some 600 terms has been furnished to all of the subcommittee members for review. It has been suggested that the final report be a joint effort of the ASCE and ASTM committees. It is planned that another subcommittee meeting will be held during the Annual Meeting of the Society to complete this review.

Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigation (J. O. Osterberg, chairman).—The scope of the subcommittee activities has been broadened, as noted in title. The subcommittee has prepared three methods: (*1*) split-tube sampling of soils, (*2*) thin-walled tube sampling of soils, and (*3*) proposed method of boring and sampling for soil investigations of embankment material. Additional work has been carried out on these proposed methods during the past year and they will be submitted to the committee shortly.

Subcommittee R-3 on Physical Characteristics of Soils (Harold Allen, chairman).—Section A has been working on a revision of Standard Method of Test for Mechanical Analysis of Soils (D 422 – 51). Considerable exchange of research information has been necessary in the preparation of a satisfactory revision of the method. A letter ballot on the adoption as standard of the Tentative Method of Test for the Amount of Material in Soils Finer than the No. 200 Sieve (D 1140 – 50 T) has been submitted to the subcommittee membership.

Section B has submitted letter ballots on revised versions of the following tests to the subcommittee: (1) Standard Method of Test for the Liquid Limit of Soils (D 423 – 39), and (2) Standard Method of Test for Plastic Limit and Plasticity Index of Soil (D 424 – 39).

Section C has prepared a revision of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T).

Subcommittee R-4 on Physical Properties of Soils (E. S. Barber, chairman).—Study of a test procedure for permeability is continuing. A Symposium on Permeability is being organized for the 1954 Annual Meeting.

Subcommittee R-5 on Structural Properties of Soils (D. M. Burmister, chairman).—Section B has circularized its members regarding the possibility of a cooperative program of testing certain standardized soils by the direct shear method. Inasmuch as most of the members were not in a position to undertake research at this time, the cooperative program was abandoned for the time being. Consideration is being given to the possibility of developing a proposed standard testing machine and procedure without the benefit of the cooperative program.

Subcommittee R-6 on Physico-Chemical Properties of Soils (R. C. Mielenz, chairman) has discussed plans for informal conferences or seminars on techniques and procedures of investigating the physico-chemical properties of soils. The most appropriate topics for early conferences are: (*1*) differential thermal analyses, (*2*) X-ray diffraction analyses, (*3*) petrographic analyses, and (*4*) chemical analyses. It is proposed that these seminars be coordinated with activities of other groups interested in clay and clay technology. The conferences will emphasize new developments and procedures with specific reference to their limitations.

Subcommittee R-9 on Dynamic Properties of Soils (R. K. Bernhard, chairman).—A list of publications referring to soil dynamics is available from Society headquarters. The subcommittee is working on a nomenclature of terms used in the field of soil dynamics. This subcommittee is preparing a Symposium on Soil Dynamics to be given at the 1953 Annual Meeting of the Society.[1]

Subcommittee R-10 on Bearing Tests of Soil In-Place (L. A. Palmer, chairman).—Section B on Pile Load Bearing Tests (A. E. Cummings, chairman) is preparing a Symposium on Lateral Pile Load Tests, which will be held at the 1953 Annual Meeting.[2]

Joint Subcommittee D-4, D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman) completed its work on the new Tentative Specifications for Materials for Soil-Aggregate Sub-base, Base, and Surface Courses (D 1241 – 52 T), as noted earlier in this report. Other work consisted of reviewing existing specifications.

This report has been submitted to letter ballot of the committee, which consists of 97 voting members; 83 members returned their ballots, of whom 80 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. Kilcawley,
Chairman.

W. G. Holtz,
Secretary.

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[1] To be issued as separate publication *ASTM STP No. 156.*

[2] To be issued as separate publication *ASTM STP No. 154.*

REPORT OF COMMITTEE D-19

ON

INDUSTRIAL WATER*

Meetings of Committee D-19 and all of its subcommittees were held on June 25 to 27, 1952, in New York, N. Y., and on January 26 to 28, 1953, in St. Louis, Mo. An additional meeting of Subcommittee I, Advisory, was held on October 21, 1952, in Pittsburgh, Pa.

A Symposium on the Continuous Analysis of Industrial Water and Industrial Waste Water, sponsored by the committee, was presented on June 24, 1952, in New York, N. Y. The papers and discussions have been published in *ASTM Special Technical Publication No. 130*.

The Manual on Industrial Water, a committee project, has been published as *ASTM Special Technical Publication No. 148*. The methods of the committee included in the Manual have been reprinted from the 1952 Book of ASTM Standards, Part 7.

A Guide for the Administration and Operation of Committee D-19 on Industrial Water has been printed for the use of the members of the committee.

An informal Task Group for Uniformity of Standard Methods for the Examination of Water was organized on January 27, 1953, in St. Louis, Mo. Details are given later in this report under the Activities of Subcommittees, Subcommittee I, Advisory.

Members of Committee D-19 serve as Society representatives on a number of other organizations as follows:

Executive Committee, Joint Research Committee on Boiler Feedwater Studies—C. H. Fellows and L. D. Betz, the latter succeeding F. N. Speller. The report of the activities of the Joint Research Committee has been published in the ASTM BULLETIN.[1] The statement includes an outline of the forthcoming proposed research on the corrosion of steel by boiler water.

Subcommittee on Standard Methods for the Examination of Water and Sewage, of the American Public Health Association—Frank E. Clarke and Roy F. Weston.

Committee on Standard Methods of Sewage and Industrial Waste Analysis, of the Federation of Sewage and Industrial Waste Associations—L. D. Betz.

Subcommittee for the Care of Pressure Vessels in Service and its Chemical Section, of the ASME Boiler Code Committee—R. A. Lorenzini, succeeding Max Hecht.

Corrosion Research Council of the Engineering Foundation—Frank N. Alquist.

At present 114 members are serving on Committee D-19.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Examination of Water-Formed Deposits by Chemical Microscopy (D 1245 – 52 T), and

Test for Bromide Ion and Iodide Ion in Industrial Water (D 1246 – 52 T), both prepared by Subcommittee IV.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] ASTM BULLETIN, No. 188, February, 1953, p. 22.

Revision of Tentative Specifications for:

Reagent Water (D 1193 - 51 T), prepared by Subcommittee II.

Revision and Reversion to Tentative of Standard Method of:

Sampling Industrial Water (D 510 - 49), prepared by Subcommittee III.

These recommendations were accepted by the Standards Committee on September 5, 1952, and the new and revised tentatives appear in the 1952 Book of ASTM Standards, Part 7.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information the following three proposed methods, as appended hereto:

Odor of Industrial Waste Water, including a classification of odors,[2]
pH of Industrial Waste Water,[2] and
Residue on Evaporation of Filtered Industrial Waste Water.[3]

NEW TENTATIVES

The committee recommends for publication as tentative the following four methods and scheme for analysis, as appended hereto:[4]

Tentative Methods of Test for:

Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water,
Residual Chlorine in Industrial Water,
Nitrite Ion in Industrial Water, and
Sulfides in Industrial Waste Water.

Tentative Scheme for:

Analysis of Industrial Water.

REVISION OF TENTATIVE

The committee recommends that the Tentative Method of Test for Hardness in Industrial Water (**D 1126 - 50 T**) be revised as appended hereto.[5]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the revision of the Standard Definitions of Terms Relating to Industrial Water (**D 1129 - 51**), as follows:

Add the following new definitions:

Biological Tests.—Examination for the purpose of determining the presence of, numbers of, or effects of any organism present in industrial water, or for the purpose of identifying such organisms. (D 510)

Chemical Oxygen Demand (*C.O.D.*).—The amount of oxygen, expressed in parts per million, consumed under specified conditions in the oxidation of the organic and oxidizable inorganic matter contained in an industrial waste water, corrected for the influence of chlorides.

Residual Chlorine (*Chlorine Residual*).—The amount of available chlorine present in industrial water at any specified period, subsequent to the addition of chlorine.

Total Chlorine Residual.—Total amount of chlorine residual present, without regard to type.

Free Available Chlorine Residual.—Residual consisting of hypochlorite ions (OCl), hypochlorous acid (HOCl), or a combination thereof.

Combined Available Chlorine Residual.—Residual consisting of chlorine combined with ammonia nitrogen or nitrogenous compounds.

Threshold Odor Number.—The greatest dilution with odor-free water that gives a perceptible odor.

In the definition of "Hardness," revise the second paragraph of the note to read as follows: "For industrial water, hardness was originally measured by the amount of soap required to produce a stable lather. Measurement is usually made on a water sample, the alkalinity of which has been adjusted to eliminate the effect of hydrogen ions." Also, in the third paragraph of the present note, insert the word "soap" ahead of the initial word "hardness."

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[2] This method was accepted by the Society as tentative on September 9, 1953, and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

[3] See p. 475.

[4] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

[5] The revised method was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

Revise the heading "Dissolved Solids" to read "Dissolved Solids (Dissolved Matter)."

Revise the heading "Suspended Solids" to read "Suspended Solids (Suspended Matter)."

Revise the heading "Total Solids" to read "Total Solids (Total Matter)."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following twelve tentatives be continued as tentative without revision, pending further study:

Tentative Methods of Test for:

Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Water (D 513 - 50 T),
Silica in Industrial Water (D 859 - 50 T),
Dissolved Oxygen in Industrial Water (D 888 - 49 T),
Sampling of Steam (D 1066 - 49 T),
Acidity and Alkalinity in Industrial Water (D 1067 - 51 T),
Iron in Industrial Water (D 1068 - 49 T),
Suspended and Dissolved Solids in Industrial Water (D 1069 - 49 T),
Electrical Conductivity of Industrial Water (D 1125 - 50 T),
Sodium and Potassium in Industrial Water (D 1127 - 50 T),
Identification of Types of Microorganisms in Industrial Water (D 1128 - 50 T),
Chloroform-Extractable Matter in Industrial Water (D 1178 - 51 T), and
Fluoride Ion in Industrial Water (D 1179 - 51 T).

The literature references supplying supporting data for the preceding recommendations, together with other credits and acknowledgements, are tabulated in Appendix I to this report.[6]

The recommendations appearing in this report have been submitted to letter vote of the committee, the results of which will be reported at the Annual Meeting of the Society.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Advisory (Max Hecht, chairman).—The Task Group on Improvement of the Committee Structure, organized October, 1952, held an initial meeting on January 27, 1953. The group includes R. C. Adams, chairman, and the chairmen of the subcommittees.

The initial proposal now being studied is the reorganization of the committee into seven subcommittees, including an Advisory Subcommittee. It is intended to assign all of the current and forseeable activities of the committee to the following proposed subcommittees:

Literature and Specifications,
Methods of Sampling,
Methods of Chemical Analysis,
Methods of Physical Determinations,
Testing Procedures, and
Performance Tests.

A task group for the proposed Committee D-19 Achievement Award was appointed with the following personnel: L. Drew Betz, chairman, Frank N. Alquist, and S. K. Love. It will submit recommendations on a proposal that an annual committee award be made to a selected member for outstanding activity.

The informal Task Group for Uniformity of Standard Methods for the Examination of Water, organized January 27, 1953, includes in its personnel the following organizations and members: F. W. Gilcreas and Paul Kabler, representing the A.P.H.A. Subcommittee on Standard Methods for the Examination of Water and Sewage; Gail P. Edwards and C. C. Ruchhoft, representing the F.S.I.W.A. Committee on Standard Methods of Sewage and Industrial Waste Analysis; A. A. Berk, of the A.S.M.E.

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[6] See p. 470.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Power Test Code Committee; Frank E. Clarke and Roy F. Weston, the ASTM representatives to the A.P.H.A. Subcommittee on Standard Methods; and L. Drew Betz, the ASTM representative to the F.S.I.W.A. Committee on Standard Methods. *Ex-officio* members are W. D. Hatfield, chairman of the F.S.I.W.A. Committee on Standard Methods, and Claude K. Rice, Standards Advisor to Committee D-19.

The Task Group, serving in an advisory capacity, will review published methods and suggest uniformity of title, definitions of terms, reagents, procedure, etc.; and it will supply the desired cross references for succeeding publications. The suggestions, which are limited to the substance of methods, will then be referred to each of the participating organizations.

Membership in the personnel of the Task Group may be expanded to include participation by other interested organizations.

The D-19 Standards Advisor, Claude K. Rice, has reviewed editorially all material, other than papers and the Manual on Industrial Water, that the committee has submitted to the Society during the past year for publication. This has resulted in a substantial improvement in the editorial quality of the material as submitted.

Bruce W. Dickerson has been elected by the Advisory Subcommittee to serve as Secretary, succeeding R. T. Sheen, resigned.

After many years of valuable service to Section B of Subcommittee II, Section C of Subcommittee IV, and the Committee as a whole, C. E. Imhoff has resigned his connections with the committee. Committee D - 19 and Subcommittees II and IV have gone on record as expressing their appreciation of the services Mr. Imhoff has rendered.

Subcommittee II on Definitions and General Specifications (S. K. Love, chairman):

Section A, Manual (R. C. Adams, chairman) completed the Manual on Industrial Water, mentioned earlier in this report. Mr. Adams was not only responsible for the final revised manuscripts but also handled the reviews of proofs and other details in connection with publication of the Manual. Richard D. Hoak has been appointed chairman of this section to succeed R. C. Adams.

Section B on Terminology and Nomenclature (R. E. Price, chairman) reviewed the definitions included in the methods that recently have been published by the Society or that are appended to this report and has prepared the revision of the Standard Definitions of Terms Relating to Industrial Water (D 1129 - 51), which is being recommended for immediate adoption.

Section D on General Specifications (R. J. Austin, chairman) is currently studying the inclusion of requirements for organic matter in the Tentative Specifications for Reagent Water (D 1193 - 52 T).

Subcommittee III on Methods of Sampling (O. M. Elliot, chairman) is studying the Tentative Specifications for Equipment for Sampling Industrial Water and Steam (D 1192 - 51 T) with regard to the use of plastic containers.

Further study is being given to the Tentative Methods of Sampling Steam (D 1066 - 49 T) to include procedures for sampling water and steam at subatmospheric pressures.

Subcommittee IV on Methods of Analysis (F. E. Clarke, chairman) has prepared the proposed Tentative Methods of Test for Residual Chlorine in Industrial Water, and for Nitrite Ion in Industrial Water, and the proposed Tentative Scheme for Analysis of Industrial Water, appended hereto.[4]

Section A, Analytical (F. E. Clarke,

chairman) has made progress in the preparation of proposed new and revised methods for organic matter, sulfite, hydrogen, acidity and alkalinity, carbon dioxide, etc., orthophosphates, etc., suspended and dissolved solids, and dissolved oxygen. The proposed revisions to the dissolved oxygen method are based on the results secured at a comprehensive service test clinic held in Baltimore, July, 1952. The results from this clinic are reported in the paper by Riedel presented at this meeting.[8] Exploratory work is being continued on methods for ammonia, cupric ion, color, and turbidity, and on proposed revisions to the Standard Method of Test for Sulfate Ion in Industrial Water (D 516 - 49).

Section B on Electrometric Methods (Robert Rosenthal, chairman) is preparing a proposed method for oxidation-reduction potential in water.

Section C on Instrumental Methods (A. O. Walker, chairman) has made progress on the preparation of proposed methods for spectroscopic and for flame photometric examination for constituents of industrial water. It is studying a proposed method for the polarographic examination of water.

Section D on Chemical Analysis of Water-Formed Deposits (F. U. Neat, chairman) has prepared a second draft of a proposed method for the chemical analysis of water-formed deposits. This method will be provided to the members who are participating in a series of round-robin tests for securing verification of the method. The data secured in this test will provide for the statement of precision and accuracy of results. The method will then be reviewed and the data evaluated.

Subcommittee VI on Corrosivity Testing (Frank N. Alquist, chairman) reports material progress on the two projects mentioned below.

The Task Group on Service Testing of Tubular Metallic Materials is continuing the collection of data secured with the apparatus. It was described sketchily in the 1952 Report of Committee D-19 under the activities of Subcommittee VI on Methods of Testing. The subcommittee desires from the cooperators detailed data on the chemical composition of and the identification of water-formed deposits, supplementing the photographic record of the deposits.

The Task Group on the Turner Test Gage is assembling data secured by operators who have made use of this device, for the purpose of development of a test procedure. A paper containing this data is being prepared for presentation at one of the meetings of the Joint Research Committee on Boiler Feedwater Studies in 1953.

Subcommittee VII on Industrial Waste Water (L. Drew Betz, chairman) has prepared the proposed Tentative Methods of Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) in Industrial Waste Water and for Sulfides in Industrial Water, appended hereto.[4] The subcommittee has also prepared the proposed Methods of Test for Odor of Industrial Waste Water,[2] pH of Industrial Waste Water,[2] and Residue on Evaporation of Filtered Industrial Waste Water,[3] which are recommended for publication as information only, as appended hereto. A listing of technical associations, trade associations, and regulatory bodies interested in waste waters has been revised by R. F. Weston and will be distributed in the near future.

Section A on Critical Constituents on Industrial Waste Water (S. A. Braley, chairman) is considering listing the critical constituents by technical operations in place of industries. The subcommittee members will be circularized for comment and suggestions on this proposed change and for critical constituents under the proposed classifications.

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[8] See p. 1103.

Section B on Analytical Methods (R. F. Weston, chairman) expects to complete proposed methods for oily matter in industrial waste water and suspended solids in industrial waste water prior to the Annual Meeting. Substantial progress is reported on the preparation of proposed methods of test for the following: turbidity and color, acidity and alkalinity, chlorine requirements, toxicity to aquatic life, phenolic compounds, cyanide, and specific gravity. Studies have been undertaken on the following new projects: mercaptans, assigned to the Sulfur Compounds Task Group; biological oxygen demand (B.O.D.) and low concentrations of chemical oxygen demand (C.O.D.), assigned to the C.O.D. Task Group; infrared method for oil, assigned to the Oily Matter Task Group. Studies will also be started on nitrogen bases and taste.

Section C on Gaging and Sampling of Industrial Waste Water and Preservation of Samples has abandoned the task group on preservation of samples. The information on preservation when required for a specific test is to be incorporated in the method for that test.

Section D on Reporting Analysis of Industrial Waste Water (W. W. Hodge, chairman) is conducting further studies on reporting the results of analysis of industrial waste water. It has recommended that the Proposed Method of Test for Odor be published as information, as appended.[2]

This report has been submitted to letter ballot of the committee, which consists of 114 members; 96 have returned their ballots, of whom 71½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. T. SHEEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Chlorine Requirements of Industrial Water and Industrial Waste Water (D 1291 - 53 T),
Test for Odor of Industrial Waste Water (D 1292 - 53 T), and
Determination of pH of Industrial Waste Water (D 1293 - 53 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new tentative methods appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

APPENDIX I

SUPPORTING DATA AND PERTINENT LITERATURE REFERENCES RELATING TO NEW AND REVISED METHODS OF TESTING INDUSTRIAL WATER

Supporting data and pertinent literature references relating to the various new and revised methods of testing industrial water included in the Report of Committee D-19, are given in this Appendix.

LITERATURE REFERENCES RELATING TO PROPOSED METHOD OF TEST FOR ODOR OF INDUSTRIAL WASTE WATER

Literature References:

(1) "Standard Methods for the Examination of Water and Sewage," Ninth Edition, American Public Health Association. New York, N. Y. (1946).

(2) C. H. Spaulding, *American Journal of Public Health*, Vol. 21, p. 1038 (1931).

(3) J. R. Baylis, *Water Works and Sewage*, Vol. 79, p. 425 (1932).

(4) G. M. Fair, *Journal of New England Water Works Association*, Vol. 47, p. 248 (1933).

(5) N. Hulbert and D. Feben, *Journal of American Water Works Association*, Vol. 33, 1945 (1941).

(6) C. H. Spaulding, *Journal of American Water Works Association*, Vol. 34, p. 877 (1942).

(7) E. C. Crocker, "An Odor Scheme," Private Communication to F. N. Alquist.

(8) E. C. Crocker, "Flavor," Published by McGraw-Hill Book Co. (1945).

(9) Cargille Scientific, Inc. "Scientific Odor Control." Published by Cargille Scientific, Inc., 118 Liberty St., New York 6, N. Y.

SUPPORTING DATA RELATING TO PROPOSED METHOD OF TEST FOR RESIDUE ON EVAPORATION OF FILTERED INDUSTRIAL WASTE WATER

Supporting Data:

This procedure is based on information contained in the literature, such as the Tentative Methods of Test for Suspended and Dissolved Solids in Industrial Water (ASTM Designation: D 1069),[1] and the Standard Methods for the Examination of Water and Sewage, Am. Public Health Assoc., Ninth Edition, pp. 145–146 (1946). The broad coverage required for the great variety of industrial waste water has been recognized. Results of cooperative work of D-19 Subcommittee VII, Section B, are shown in Table I.

LITERATURE REFERENCES RELATING TO PROPOSED TENTATIVE METHOD OF TEST FOR THE CHEMICAL OXYGEN DEMAND (DICHROMATE OXYGEN DEMAND) OF INDUSTRIAL WASTE WATER

Literature References:

(1) W. A. Moore, R. C. Kroner, and C. C. Ruchhoft, "Dichromate Reflux Method for Determination of Oxygen Consumed," *Analytical Chemistry*, Vol. 21, p. 953 (1949).

(2) W. A. Moore, F. J. Ludzack, and C. C. Ruchhoft, "Determination of Oxygen consumed Values of Organic Wastes, A Comparative Study of Methods," *Analytical Chemistry*, Vol. 23, p. 1297 (1951).

SUPPORTING DATA AND LITERATURE REFERENCES RELATING TO PROPOSED TENTATIVE METHOD OF TEST FOR NITRITE ION IN INDUSTRIAL WATER

Supporting Data:

A1. (*a*) To determine the minimum concentration of ferric iron which gives color interference in the determination of nitrite nitrogen with sulfanilic acid and alpha naphthylamine, see the readings (Table II) made at 545 mμ with 0.1 ppm nitrite nitrogen and increments of $FeCl_3 \cdot 6H_2O$, and also the data shown in Table III.

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[1] 1952 Book of ASTM Standards, Part 7.

TABLE I.—RESULTS FOR RESIDUE ON EVAPORATION OF SAMPLES OF WASTE WATER.

Nature of Waste Water	Parts per Million			
	Test 1		Test 2	
Discharge from waste treatment plant. Wastes from pickling and plating operation. Size of sample evaporated was 2000 g, results in duplicate	252		250	
	(1)	(2)	(3)	(4)
Miscellaneous industrial and domestic wastes from high-polluted stream. Size of sample evaporated was 100 g, sampled four separate days, results in duplicate	317 317	543 552	523 522	332 331
	Effluent No. 1		Effluent No. 2	
Brackish once-through cooling water. Size of sample used, g:				
5	17400 17900 17700		20700 20900 21600	
10	18500 18600 18300		21300 20700 22000	
50	18200 19600 21000		23000 26800 22400	

TABLE II.—EFFECT OF FERRIC IRON ON READINGS AT 545 mμ.

Nitrite Nitrogen, ppm	Ferric Iron, ppm	Optical Density
	0	0.230
	0	0.230
0.1	1	0.230
	2.5	0.230
	5	0.245
	7.5	0.245
	10.0	0.245
	25	0.282
	50	0.295
	1	0.225
	2	0.235
0.1	3	0.235
	4	0.238
	5	0.238
	6	0.238
	7	0.240
	8	0.245
	9	0.245
	10	0.245

(*b*) *Conclusions:*

(1) For nitrite nitrogen concentrations below 0.05 ppm, 4 ppm ferric iron cause an increase in optical density.

(2) For nitrite nitrogen concentrations between 0.05 ppm and 0.2 ppm, 6 ppm ferric iron cause interference.

(3) At and below 3 ppm ferric iron, there is no significant increase in optical density at any point on the nitrite curve, if the readings are made when the nitrite color has developed 10 min, as directed.

TABLE III.—MINIMUM CONCENTRATIONS OF FERRIC IRON THAT GIVE COLOR INTERFERENCE.

Nitrite Nitrogen, ppm	Optical Density				
	Fe = 0	1 ppm Fe	3 ppm Fe	5 ppm Fe	7 ppm Fe
0.001	0.013	0.016	0.015	0.027	0.033
0.002	0.016	0.020	0.022	0.027	0.035
0.005	0.028	0.027	0.030	0.036	0.041
0.007	0.032	0.030	0.033	0.045	0.055
0.010	0.038	0.045	0.038	0.055	0.062
0.020	0.065	0.063	0.065	0.075	0.080
0.050	0.127	0.128	0.128	0.132	0.140
0.100	0.235	0.232	0.232	0.235	0.245
0.200	0.435	0.430	0.435	0.435	0.450
0.300	0.62	0.60	0.61	0.62	0.62

TABLE IV.—INCREASE IN OPTICAL DENSITY DUE TO FERRIC IRON.

Ferric Iron, ppm	Optical Density, $FeCl_3 \cdot 6H_2O$ Only	$FeCl_3 \cdot 6H_2O$ plus Nitrite Reagents	
		I	II
1	0.000	0.010	0.015
2.5	0.002	0.013	...
5	0.002	0.023	0.020
7.5	0.010	0.023	...
10	0.014	0.023	0.025
25	0.025	0.048	0.055
50	0.030	0.070	0.073
75	0.030	0.088	0.090
100	0.030	0.123	0.128
150	0.025	0.220	...
200	0.023	0.290	0.230
250	0.020	0.390	...

(4) At concentrations above 3 ppm ferric iron, there is a gradual increase in optical density due to the color formed by iron plus the nitrite reagents. The interference value of this color is inversely proportional to the density of the nitrite color, as shown in Table IV.

Literature References:

(1) M. L. Ilosvay, "Nitrous Acid in the Saliva and in Exhaled Air," *Bull. de la Soc. Chem.*, Ser. 3, Vol. 2, p. 388 (1889).

TABLE V.—DETERMINATION OF TOTAL HARDNESS BY NON-REFEREE METHOD A OF METHODS D 1126—PRECISION STUDY.

SAMPLE NO. 1—DILUTED CLEVELAND CITY WATER

	Total Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	29.5	29.8
2	29.5	29.5
3	29.4	29.8
4	29.3	29.4
5	29.4	29.2
6	29.5	29.4
7	29.4	29.2
8	29.5	29.3
9	29.4	29.2
10	29.5	29.2
Average	29.44	29.40
Standard Deviation$_{10}$	0.06	0.22
Standard Deviation$_{20}$	0.17	

Precision = 0.57 per cent of hardness present

SAMPLE NO. 2—BOILER FEEDWATER

	Total Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	54.4	54.4
2	54.6	54.0
3	54.2	54.2
4	54.2	54.0
5	54.4	54.0
6	54.4	54.2
7	54.4	54.2
8	54.2	54.2
9	54.4	54.0
10	54.4	54.0
Average	54.36	54.12
Standard Deviation$_{10}$	0.12	0.13
Standard Deviation$_{20}$	0.17	

Precision = 0.31 per cent of hardness present

SAMPLE NO. 3—COOLING WATER FROM AIR-CONDITIONING SYSTEM (COLORED WITH ORGANIC MATTER)

	Total Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	118.0	117.2
2	118.5	118.0
3	118.5	117.6
4	117.5	117.6
5	118.0	117.2
6	118.0	117.6
7	117.5	117.2
8	118.5	117.6
9	117.5	117.6
10	117.5	117.2
Average	117.95	117.48
Standard Deviation$_{10}$	0.42	0.26
Standard Deviation$_{20}$	0.42	

Precision = 0.36 per cent of hardness present

SAMPLE NO. 4—DILUTED LAKE ERIE WATER (HIGH TURBIDITY)

	Total Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	67.0	67.6
2	67.8	67.4
3	66.8	67.4
4	67.4	67.2
5	67.0	67.4
6	66.8	67.4
7	67.0	67.2
8	66.8	67.2
9	66.8	67.0
10	66.8	67.2
Average	67.02	67.30
Standard Deviation$_{10}$	0.32	0.16
Standard Deviation$_{20}$	0.29	

Precision = 0.42 per cent of hardness present

(2) R. S. Weston, "Notes on the Determination of Nitrogen as Nitrites in Waters," *Journal American Chemical Society*, Vol. 27, p. 281 1905).

(3) "Standard Methods for the Examination of Water and Sewage," Ninth Edition, American Public Health Association, New York, N. Y., pp. 71–72 (1946).

LITERATURE REFERENCES RELATING TO PROPOSED METHOD OF TEST FOR SULFIDES IN INDUSTRIAL WATER

Literature References:

(1) R. D. Pomeroy, "The Determination of Sulfides in Sewage," *Sewage Works Journal*, Vol. 8, P. 572 (1936); and Vol. 13, p. 498 (1941).

(2) R. D. Pomeroy, *The Petroleum Engineer*, Sept., 1944, p. 156.

TABLE VI.—DETERMINATION OF CALCIUM HARDNESS BY D 1126—NON-REFEREE METHOD A.

SAMPLE NO. 1—DILUTED CLEVELAND CITY WATER

	Calcium Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	21.8	21.5
2	21.9	21.6
3	21.8	21.8
4	22.0	21.7
5	22.0	21.7
6	21.9	21.6
7	21.9	21.6
8	22.0	21.8
9	22.0	21.7
10	22.0	21.8
Average	21.93	21.68
Standard Deviation$_{10}$	0.08	0.10
Standard Deviation$_{20}$	0.15	

Precision = 0.69 per cent of CaCO$_3$ present

SAMPLE NO. 2—BOILER FEEDWATER

	Calcium Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	40.7	40.0
2	40.9	40.1
3	40.9	40.3
4	40.8	40.4
5	40.8	40.4
6	40.9	40.3
7	40.9	40.3
8	40.9	40.2
9	40.9	40.4
10	40.8	40.4
Average	40.85	40.28
Standard Deviation$_{10}$	0.07	0.13
Standard Deviation$_{20}$	0.32	

Precision = 0.71 per cent of CaCO$_3$ present

SAMPLE NO. 3—COOLING WATER FROM AIR CONDITIONING SYSTEM (COLORED WITH ORGANIC MATTER)

	Calcium Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	84.8	83.5
2	84.4	84.3
3	84.6	84.3
4	84.6	84.7
5	84.8	84.7
6	85.0	84.7
7	84.8	84.7
8	84.8	85.1
9	85.0	84.7
10	84.6	85.1
Average	84.74	84.58
Standard Deviation$_{10}$	0.18	0.31
Standard Deviation$_{20}$	0.33	

Precision = 0.39 per cent of CaCO$_3$ present

SAMPLE NO. 4—DILUTED LAKE ERIE WATER (HIGH TURBIDITY)

	Calcium Hardness, ppm CaCO$_3$	
	Analyst A	Analyst B
1	49.7	49.1
2	49.8	49.4
3	49.7	49.5
4	49.8	49.6
5	49.6	49.4
6	49.4	49.4
7	49.5	49.5
8	49.6	49.4
9	49.5	49.4
10	49.7	49.5
Average	49.63	49.42
Standard Deviation$_{10}$	0.13	0.13
Standard Deviation$_{20}$	0.16	

Precision = 0.32 per cent of CaCO$_3$ present

SUPPORTING DATA AND LITERATURE REFERENCES RELATING TO REVISED TENTATIVE METHODS OF TEST FOR HARDNESS IN INDUSTRIAL WATER

Supporting Data for Non-Referee Method A:

Data on the accuracy of Non-Referee Method A appears in papers by J. D. Betz and C. A. Noll.[2] Precision data for Non-Referee Method A was obtained from four different waters, using two operators. These data are listed in Tables V and VI.

Literature References for Non-Referee Method A:

(**1**) J. D. Betz and C. A. Noll, "Total Hardness Determination by Direct Colorimetric Titration," *Journal American Water Works Association*, Vol. 42, January, 1950, pp. 49–56.

(**2**) J. D. Betz and C. A. Noll, "Further Studies With the Direct Colorimetric Hardness Titration," *Journal American Water Works Association*, Vol. 42, August, 1950, pp. 749–754.

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[2] See literature references (**1**) and (**2**) applying to this Non-Referee Method A.

(3) W. Biedermann and G. Schwarzenbach, "The Complexometric Titration of Alkaline Earths and Some Other Metals with Eriochrome Black T," *Chimia*, Vol. 2, p. 56 (1948).

(4) J. J. Connors, "Advances in Chemical and Colorimetric Methods," *Journal American Water Works Association*, Vol. 42, January, 1950, pp. 33–39.

(5) H. Diehl, C. A. Goetz, and C. C. Hach, "The Versenate Titration for Total Hardness," *Journal American Water Works Association*, Vol. 42, January, 1950, pp. 40–48.

(6) C. A. Goetz, T. A. Loomis, and H. Diehl, "Total Hardness in Water," *Analytical Chemistry*, Vol. 22, June, 1950, pp. 798–799.

(7) V. M. Marcy, "New Water Hardness Test is Faster and Gives More Accurate Results," *Power*, Vol. 94, January, 1950.

(8) V. M. Marcy, "Rapid Test for Calcium Hardness," *Power*, Vol. 94, June, 1950, pp. 92–93.

(9) G. Schwarzenbach and H. Ackerman, "Complexons V. Ethylenediaminetetraacetic Acid," *Helvetica Chimica Acta*, Vol. 30, p. 1798 (1947).

(10) G. Schwarzenbach, W. Biedermann, and F. Bangerter, "Complexons VI. New Simple Titrating Methods for Determining the Hardness of Water," *Helvetica Chimica Acta*, Vol. 29, p. 811 (1946).

APPENDIX II

PROPOSED METHOD OF TEST FOR RESIDUE ON EVAPORATION OF FILTERED INDUSTRIAL WASTE WATER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope and Application

1. (*a*) This method covers a procedure for determining the residue on evaporation of filtered industrial waste water, free of suspended matter.

(*b*) This method is intended to be used as a non-referee method of test for industrial waste water containing a wide range of dissolved matter.

Principle of Method

2. Dissolved matter is determined by weighing the residue obtained after evaporating the sample and drying it at 103 C.

NOTE 1.—The suggested drying temperature of 103 C does not necessarily provide complete dehydration of all residues. It is permissible to use other temperatures and time of drying by mutual agreement. In all cases the drying temperature shall be specified in reporting results. Strict adherence to drying time should be observed.

Definitions

3. (*a*) The term "dissolved matter" in this method is defined in accordance with the Standard Definitions of Terms Relating to Industrial Water (ASTM Designation: D 1129),[2] as follows:

Dissolved Solids (*Dissolved Matter*).—The dried residue from evaporation of the filtrate after separation of suspended solids.

(*b*) For definitions of other terms used in this method, refer to Definitions D 1129.

Interferences

4. Some waste waters contain dissolved matter or salts that hydrolyze or volatilize upon evaporation of the sample and subsequent drying at 103 C. For example, $(NH_4)_2CO_3$ decomposes below this temperature. Some liquids, such as glycerol and H_2SO_4, will not dehydrate at 103 C, and therefore remain with the solids.

Apparatus

5. (*a*) *Sample Reservoir*.—A chemically resistant glass container of 1- to 4-liter capacity. Smaller reservoirs may be used for waters with high concentrations.

(*b*) *Evaporating Dish*.—A straight-walled platinum dish approximately 80- to 100-mm in diameter and of 200-ml capacity.

(*c*) *Heater*.—A controlled electric hot-plate, infrared lamp, or steam bath for maintaining the temperature of the evaporating water below the boiling point.

(*d*) *Drying Oven*, electrically heated, air-circulating type, controlled to ±0.5 C.

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-19 on Industrial Water. Published as information, June, 1953.

[2] 1953 Supplement to Book of ASTM Standards, Part 7.

Sampling

6. Collect the sample in accordance with the Tentative Methods of Sampling Industrial Water (ASTM Designation: D 510).[2]

Procedure

7. (*a*) Weigh a clear sample (Note 2) of such size as to yield a residue weighing at least 25 mg. If analysis of the residue is desired, weigh sufficient sample to yield a residue weighing approximately 100 mg. Pour the sample into the sample reservoir.

NOTE 2.—The weight basis for the sample is recommended because the density of industrial waste water may vary over a wide range.

(*b*) Fill a weighed platinum dish to within $\frac{1}{4}$ in. of the top (Note 3) with water from the sample reservoir. Heat the dish by means of a hot-plate, infrared lamp, or steam bath, maintaining the sample temperature below the boiling point. Periodically add sample from the reservoir to prevent drying of the dish, until the reservoir is empty. Rinse the reservoir several times with non-referee reagent water conforming to the Tentative Specifications for Reagent Water (ASTM Designation: D 1193),[2] adding the rinsings to the contents of the evaporating dish. Evaporate the balance of the material in the dish to near dryness. Dry the dish and its contents in an oven at 103 C for 60 min, cool in a desiccator, and reweigh. Dry again for 30-min periods until the loss is no more than 4 per cent of the previous weighing (Note 4). Record the increase in weight of the dish as the weight of residue.

NOTE 3.—A porcelain dish or glass beaker may be substituted for the platinum dish if the residue is not to be analyzed.

NOTE 4.—Rapid weighing is essential, since some dried residues readily absorb moisture.

Calculation

8. Calculate the residue on evaporation of the filtered industrial waste water, in parts per million, as follows:

$$\text{Residue on evaporation, ppm} = \frac{A}{W}$$

where:

A = milligrams of residue, and

W = kilograms of sample used.

Precision and Accuracy[3]

9. (*a*) The precision is limited by balance reproducibility, oven temperature, and the nature of the residue. If the analytical balance is reproducible to 0.2 mg, careful control of test conditions should yield a precision of 5 mg, as calculated by the following formula:

$$\text{Precision, ppm} = \frac{5}{W}$$

where:

W = kilograms of sample used.

(*b*) Depending on the type of dissolved matter present, the losses or gains will cause the accuracy to range from the limits of precision to wide differences. It is therefore impossible to determine the status of accuracy in this procedure.

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[3] Supporting data pertinent to this method are given in Appendix I to the Report of Committee D-19, p. 470.

REPORT OF COMMITTEE D-20

ON

PLASTICS*

Committee D-20 on Plastics, and its subcommittees, held three meetings during the year: at New York, N. Y., on June 25 and 26, 1952; at Boston, Mass. on October 27 to 29, 1952; and at Pocono Manor, Pa. on March 23 to 25, 1953. The Advisory Committee also met at these times.

The committee was represented at the meeting of Technical Committee 61 on Plastics (ISO/TC/61) of the International Organization for Standardization held on October 2 to 4, 1952, in Turin, Italy. G. M. Kline, former chairman of Committee D-20, served as chairman of the meeting, at which nine countries were represented. The working groups agreed on a number of drafts of test methods and standards to be proposed for recommended international standards. Standardization among languages of nomenclature applying to test methods and processing characteristics is being undertaken.

The next meeting of ISO/TC/61 will be held in Stockholm, Sweden, beginning August 14, 1953. The chairmanship of the American Group for ISO/TC/61, which is assisting the ASA in the duties of the Secretariat, has been accepted by Robert Burns, succeeding E. B. Cooper.

R. M. Berg was appointed chairman of Subcommittee I on Strength Properties, succeeding M. E. Marks; H. L. McChesney was appointed chairman of Subcommittee II on Hardness Properties, to fill the vacancy created by the resignation of L. W. A. Meyer; C. H. Adams was appointed chairman of Subcommittee III on Thermal Properties, succeeding E. B. Cooper; J. W. Mighton was appointed chairman of Subcommittee V, succeeding W. C. Goggin; and H. E. Riley was appointed chairman of Subcommittee VII, succeeding D. E. Northrop.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Polyethylene Molding and Extrusion Materials (D 1248 – 52 T), and

Primary Octyl Phthalate Ester Plasticizers (D 1249 – 52 T).

Tentative Methods of Test for:

Resistance to Abrasion of Plastic Materials (D 1242 – 52 T), and

Specific Viscosity of Vinyl Chloride Polymers (D 1243 – 52 T).

Revision of Tentative Specifications for:

Phenolic Molding Compounds (D 700 – 49 T).

Revision and Reversion to Tentative of:

Standard Method of Test for Resistance of Plastics to Chemical Reagents (D 543 – 43).

These recommendations were accepted by the Administrative Committee on Standards on December 12, 1952. The new and revised specifications and methods appear in the 1952 Book of ASTM Standards, Part 6.

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*Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

REVISION OF TENTATIVES

The committee recommends revisions of the following two tentatives, as indicated:

Tentative Specifications for Nylon Injection Molding and Extrusion Compounds (**D 789 - 44 T**).[1]—These specifications have been extensively revised, as appended hereto,[2] to include one additional composition suitable for molding or extrusion, and to bring the specifications into agreement with present industrial practice.

Tentative Definitions of Terms Relating to Plastics (**D 883 - 52 T**).[1]—Revise to include the following new and revised definitions.

Alkyd Plastics.—Plastics based on resins composed principally of synthetic polymeric esters in which the recurring ester groups are an integral part of the main polymer chain. (See Polyester Plastics.)

Halocarbon Plastics.—Plastics based on resins made by the polymerization of monomers composed only of carbon and a halogen or halogens.

Metastable, adj.—An unstable condition of a plastic evidenced by changes of physical properties not caused by changes in composition or in environment.

> NOTE.—Metastable refers, for example, to the temporarily more flexible condition of some plastics after molding. No physical tests should be made while the plastic is in a metastable condition unless data regarding this condition are desired.

Polyester Plastics.—Synonymous with Alkyd Plastics.

Resin, n.—A solid, semisolid, or pseudosolid organic material which has an indefinite and often high molecular weight, exhibits a tendency to flow when subjected to stress, usually has a softening or melting range, and usually fractures conchoidally.

> NOTE: *Liquid Resin.*—An organic polymeric liquid which when converted to its final state for use becomes a solid.

Volatile Loss.—Weight loss by vaporization.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Specifications for Vinyl Chloride-Acetate Resin Plastic Sheets (**D 708 - 50**) be revised as appended hereto[3] and reverted to tentative. These specifications have been revised in order to bring them into agreement with Federal Specification L-V-351, and to provide for the addition of other grades of vinyl rigid sheets which are becoming commercially important.

ADOPTION OF TENTATIVE AS STANDARD

The committee joins with Committee D-9 on Electrical Insulating Materials in recommending that the Tentative Method of Conditioning Plastics and Electrical Insulating Materials for Testing (**D 618 - 51 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with the addition of the following sentence to Note 2 in Section 4: "Shorter conditioning times may be used for thin specimens provided equilibrium is substantially obtained."

TENTATIVES CONTINUED WITHOUT REVISION

The committee has considered all of the other tentatives not mentioned in this report which are under its jurisdiction and which have been issued for two years or longer, and recommends that they be continued in their present status until further revisions in them are made as contemplated.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (R. M. Berg, chairman).—Cooperative test studies are continuing in order to secure the information necessary to permit revision of the Tentative Method of Test for Tensile Properties of Thin Plastic Sheets and Films (D 882 – 49 T). A round-robin test program on the ski-ball impact test has been completed, and work is in progress on revision of the Tentative Method of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 47 T). A round-robin test program is also under way on methods for measurement of dynamic properties of plastics. The need for studies leading to development of test methods applicable to plastic films has become evident. Accordingly, the newly formed Section P on Stiffness will study test methods for measuring stiffness of films in place of the discharged Section I on Nonrigid Plastics. The name and intended scope of Section L has been changed from Bursting Strength to Strength Under Biaxial Stressing. Studies of particular strength properties of films will be assigned to the proper sections as needs arise.

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[1] 1952 Book of ASTM Standards, Part 6.

[2] The revised tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 6.

[3] The revised specifications were accepted by the Society as a tentative and appear in the 1953 Supplement to Book of ASTM Standards, Part 6.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Hardness (H. L. McChesney, chairman).—A subcommittee ballot on a revision of the Tentative Method of Test for Resistance of Transparent Plastics to Surface Abrasion (D 1044 – 49 T) is in progress. Revision of the Standard Method for Estimating Blocking of Plastics Sheets (D 884 – 48) is being considered. Comparative studies of the British Admiralty hardness test and comparable ASTM hardness tests are to be initiated, and test methods for evaluating scratch resistance are to be studied. The name and the intended scope of Section A was changed from Rockwell Hardness to Indentation Hardness.

Subcommittee III on Thermal Properties (C. H. Adams, chairman).—The desirability of annealing certain plastics before testing by the Tentative Methods of Test for Deformation of Plastics Under Load (D 621 – 48 T) or for Heat Distortion Temperature of Plastics (D 648 – 45 T) has been shown in studies by members of this subcommittee. Appropriate footnotes will be added to these tentatives and descriptions of satisfactory annealing techniques will be prepared for inclusion in the affected plastics specifications. Work is progressing toward preparing test methods for flammability, for distortion by heat, and for brittle temperature, which are applicable to thin films. The Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (D 746 – 52 T) has been studied, and an effort is being made to resolve differences in the results obtained with the different types of machines. Under consideration is the development of methods for determining thermal conductivity, specific heat, viscosity by parallel plate plastometer, and stress-relaxation properties.

Subcommittee IV on Optical Properties (H. K. Hammond, 3rd, chairman).—Work on a method of measurement of small color differences is continuing in cooperation with subcommittees of Committee D-1 on Paint, Varnish, Lacquer, and Related Products and Committee E-12 on Appearance. The preparation of permanent haze standards for use with the Standard Method of Test for Haze and Luminous Transmittance of Transparent Plastics (D 1003 – 52) is continuing.

Subcommittee V on Permanence Properties (J. W. Mighton, chairman).—This subcommittee reviewed a Proposed Method of Test for Measuring Water-Vapor Transmission and forwarded comments to the task group of Committee E-1 on Methods of Testing which originated it. The subcommittee is balloting on a revision of the Standard Method of Test for Water Absorption of Plastics (D 570 – 42). A new proposed Tentative Method of Test for Shrinkage of Molded and Laminated Thermosetting Plastics at Elevated Temperature has been completed and will be presented to the Society through the Standards Committee subsequent to the Annual Meeting. Study of techniques for measurement of gas permeability of plastic materials is under way. Work has started on a recommended practice for outdoor exposure in the new Section on Outdoor Exposure.

Subcommittee VI on Specifications (Lucius Gilman, chairman).—This subcommittee is preparing new specifications and revisions of a number of the present specifications. Specifications for Rubber Modified Phenolic Molding Compounds are being prepared. Revision of the Standard Specifications for Urea-Formaldehyde Molding Compounds (D 705 – 49) is under consideration. Proposed Tentative Specifications for Laminated Thermosetting Decorative Sheets have been completed and will be presented to the Society through the Standards Committee subsequent to the Annual Meeting. A revision of the Standard Specifications for Vinyl Chloride-Acetate Resin Plastic Sheets (D 708 – 50) has been completed as submitted in this report, and further early revisions are contemplated. Minor revisions of the Tentative Specifications for Methacrylate Molding Compounds (D 788 – 48 T) and Polystyrene Molding Compounds (D 703 – 49 T) are in preparation, and a round-robin testing program is being planned in order to prepare specifications for modified polystyrene plastics. Early revision of the Tentative Specifications for Ethyl Cellulose Molding Compounds (D 787 – 52 T) is under consideration. Work is in progress which will result shortly in revisions, and eventual combination into joint specifications, of the Tentative Specifications for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 – 46 T) and Nonrigid Vinyl Chloride Plastics (D 744 – 49 T). A proposed revision of the Tentative Specifications for Nylon Molding and Extrusion Composition (D 789 – 44 T) has been completed and is submitted in this report. Two new sections have been organized in this subcommittee: Section T on Specifications for Polyester Resins, and Section U on Halocarbon Plastics.

Subcommittee VII on Analytical Methods (H. E. Riley, chairman).—In addition to this tentative Method of Test for Specific Viscosity of Vinyl Chloride Polymers (D 1243 – 52 T) accepted by the Standards Committee, three other methods relating to vinyl chloride polymers, the determinations of total chlorine, of volatile content, and of particle size, will be completed soon. Further work on methods for characterizing organosols and plastisols has been discontinued by reason of lack of consumer interest. Work relating to plasticizers has continued. A method for determining heat stability of plasticizers will be completed shortly, and work on methods of determining ionizable compounds in plasticizers is in progress.

Subcommittee VIII on Research (C. R. Stock, chairman).—This subcommittee is continuing to serve in an advisory capacity on research problems. At the 50th Anniversary Meeting of the Society, in June, 1952, this subcommittee made the arrangements for the Symposium on Plastics Testing—Present and Future, sponsored by Committee D-20. Some of the papers presented reviewed the current status of tests for plastics, pointing out recognized shortcomings and needs which should be acted upon in the future. Other papers concerned current activities in a number of fields of plastics testing, which are directed toward rectifying present deficiencies. The symposium has been issued by the Society as *Special Technical Publication No. 132.*

At the October, 1952, meeting of the

subcommittee, three papers were presented, as follows:

"Data and Their Interpretation," by Prof. R. B. Finch, Massachusetts Institute of Technology.
"Accelerated Exposure Techniques," by L. Boor, U. S. Department of the Army, Philadelphia Quartermaster Depot.[5]
"A Constant Strain Stiffness Tester for Thin Plastic Films," by F. C. Dexter, Bakelite Division, Union Carbide and Carbon Corp.[6]

At the March, 1953, meeting of the subcommittee, the following three papers were presented:

"Increasing the Efficiency and Economy of Experimentation," by E. Harrington, Monsanto Chemical Co.
"The 'Dozi' Impact Strength Tester," [Demonstration] by Robert Burns, Bell Telephone Laboratories, Inc.
"Evaluation of the Boor-OQMG Snag Tester," by J. J. Lamb, L. Boor, C. Brown, and F. W. Reinhart, of the U. S. Government.

Subcommittee IX on Molds and Molding (J. L. Williams, chairman).—A round-robin testing program is being carried out to determine whether gating changes and electronic heating should be incorporated in a revision of the Tentative Recommended Practice for Transfer Molding of Specimens of Phenolic Materials (D 1046 - 49 T). Need for a new mold shrinkage test specimen is being surveyed.

Subcommittee X on Definitions, Nomenclature, and Significance of Tests (C. H. Alexander, chairman).—A number of definitions are being recommended, as outlined in this report, for inclusion in the Tentative Definitions of Terms Relating to Plastics (D 883 - 52 T). Definitions of additional terms are being developed for early consideration.

Subcommittee XIV on Conditioning (A. C. Webber, chairman).—Work is in progress to establish standard methods for measurement of relative humidity, particularly in enclosures too small to permit the use of the sling psychrometer as specified in the Standard Method of Determining Relative Humidity (D 337 - 34). The Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 51 T) is being recommended for adoption as standard. The work of this subcommittee is a joint activity with Committee D-9 on Electrical Insulating Materials.

This report has been submitted to letter ballot of the committee, which consists of 153 members; 96 members returned their ballots, of whom 84 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. K. WITT,
Chairman.

G. M. ARMSTRONG,
Secretary.

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[5] Published in ASTM BULLETIN, No. 189, April, 1953, p. 38 (TP44) as "An Improved Fadeometer," by L. Boor and S. L. Trucker.

[6] Published in ASTM BULLETIN, No. 192, September, 1953, p. 40 (TP130)

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Shrinkage of Molded and Laminated Thermosetting Plastics at Elevated Temperature (D 1299 - 53 T), and
Test for Chlorine Content of Vinyl Chloride Polymers and Copolymers (D 1303 - 53 T).

Tentative Specifications and Methods of Test for:

Laminated Thermosetting Decorative Sheets (D 1300 - 53 T).

Revision of Tentative Definition of:

Terms Relating to Plastics (D 883 - 53 T).

The Tentative Method D 1299 and Tentative Specifications and Methods D 1300 were accepted by the Standards Committee on September 9, 1953; the remaining recommendations were accepted on December 16, 1953. They all appear in the 1953 Supplement to Book of ASTM Standards, Part 6.

REPORT OF COMMITTEE D-21

ON

WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year, at Chicago, Ill., on May 20 and 21, 1953. In addition the subcommittees met in New York City on Dec. 10, 1952.

At the present time, the committee consists of 52 members, of whom 22 are classified as producers, 11 as consumers, and 19 as general interest members. There is 1 consulting member.

NEW TENTATIVES

The committee recommends for publication as tentative of the following three methods as appended hereto:[1]

Tentative Methods of Test for:

Nonvolatile Matter (Total Solids) in Water Emulsion Waxes,

Sediment in Water-Emulsion Waxes by Means of Centrifuge, and

Total Ash and Silica in Water-Emulsion Waxes

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. T. Hohnstine, chairman) is considering a long list of terms presented to them for definition by the other subcommittees. Consideration is being given to the preparation of a Glossary of Terms used by the industry.

Subcommittee II on Raw Materials (Melvin Fuld, chairman) is completing the editorial revision of two methods which have already been approved by letter ballot. These cover a suggested method for the concentration of additives in Carnauba or other vegetable waxes and the use of refractive index at 100 C as an indication of wax adulteration. These methods will be published as information only. Work is continuing on methods for the determination of dirt, and for water content of waxes, as well as on a method for the determination of the hydrocarbon content of waxes by chromatography.

Subcommittee III on Physical and Chemical Testing (Donald M. King, chairman) developed the three new tentative methods for determination of nonvolatile matter, ash content, and sediment, which are recommended earlier in this report. In addition, work is progressing on test methods for kinematic viscosity, free alkali, and stability of wax products. The Task Force on Flash Point Determination has reported that existing methods are unsatisfactory for testing wax polishes, and that several modifications of existing methods are being tried.

Subcommittee IV on Performance Tests (F. J. Pollnow, Jr., chairman).—The Section on Slip Resistance is completing its preliminary work on both the Sigler and James types of slip testers, and the methods will be referred to ballot in the subcommittee in the near future. The Section on Service Life has made considerable progress on the testing of water spot resistance of floor waxes. The method of measuring the hardness of wax films with the Sward Hardness Rocker, however, has been temporarily abandoned because of lack of correlation between laboratories. The Section on Appearance is still investigating methods of applying uniform wax films and plans a comprehensive report on the various methods which have been investigated.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentatives were accepted by the Society and appear in the 1953 Supplement to Book of ASTM Standards, Part 7.

Subcommittee V on Specifications (Wm. Joy, chairman) has reached a general agreement on the properties which should be included in a specification for industrial type water emulsion wax. Completion of this specification now awaits the development by the other subcommittees of test methods for the properties involved.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 53 voting members; 34 members returned their ballots, of whom 32 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

B. S. JOHNSON,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-22

ON

METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis held two meetings during the year: on March 5 and 6, 1952, in Cleveland, Ohio, and on November 17 and 18, 1952, at Mellon Inst., in Pittsburgh, Pa. The present membership of the committee consists of 49 members, of whom 3 are consulting members. Election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, L. C. McCabe,
Vice Chairman, F. S. Mallette,
Secretary, H. H. Schrenk.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Units (F. A. Patty, chairman).—The subcommittee devoted most of its time to definitions. About 75 terms were listed as requiring definitions now, and some 60 other suggested terms were placed in a deferred list. Tentative definitions were developed for some 20 terms, and others are being developed by correspondence. Good progress also has been made by the task groups on Abbreviations and on Symbols and Units.

Subcommittee II on Sampling Methods (L. Silverman, chairman).—A tentative report submitted by Task Group A on General Principles of Sampling is being revised for circulation to the subcommittee. Task Group B on Gases and Vapors has prepared a tentative report which is being revised. Task Group E on Stack or Collector Effluent Sampling has prepared a tentative draft, and further revisions are being made. A tentative draft on Miscellaneous Sampling by Task Group F is being revised.

Subcommittee III on Analytical Methods (J. Cholak, chairman).—The fluoride study group felt that titration employing alizarin and thorium nitrate was acceptable as to sensitivity and accuracy, but further study of certain factors is being made.

No recommendations were made regarding oxides of nitrogen, although the alpha-naphthylamine and phenol-disulfonic acid methods are favored. It was felt that these methods could be described as a stop-gap pending development of a dynamic sampling procedure.

The odor study group presented two methods for the measurement of odors. One was a dilution method and the other, designed to supplement the dilution method, employed activated carbon adsorption and identification of pollutant by analysis. The methods, after revision, will be submitted to all members of Subcommittees I and III for letter ballot approval.

Subcommittee IV on Instrumentation (M. D. Thomas, chairman).—A draft of a report on the sulfur dioxide analyzer will be submitted to the subcommittee for approval. Other methods under consideration are the spot method for dirt, directional dust-fall collection, oxidant recorder, and aerosol analysis by light-scattering.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. C. McCABE,
Chairman.

H. H. SCHRENK,
Secretary.

REPORT OF COMMITTEE D-23

ON

CELLULOSE AND CELLULOSE DERIVATIVES*

Certain groups of Committee D-23 on Cellulose and Cellulose Derivatives met, in the course of organization, on February 21, 1952, in New York City during the Annual Meeting of the Technical Association of the Pulp and Paper Industry. On September 19, 1952, the subcommittees and the committee met at Atlantic City, N. J., during the annual meeting of the American Chemical Society. The subcommittees completed organizational details and general program plans. A new Subcommittee on Statistics was established at this time.

At the meeting of the committee, the following officers were elected for the term ending in 1954:

Chairman, Forrest A. Simmonds,
Vice-Chairman, Leo B. Genung,
Secretary, Walter W. Becker.

On February 19, 1953, the Executive Subcommittee held an open meeting at New York City in conjunction with the Annual Meeting of the Technical Association of the Pulp and Paper Industry. The business transacted was chiefly details of organization and procedure.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Cellulose (R. M. Levy, chairman) is, understandably, a very large group. It is presently made up of eleven task groups. These and the respective chairmen are:

Absorption and Color, James A. Mitchell;
Alpha, Beta, and Gamma Celluloses, E. E. Hembree;
Ash Constituents, J. E. Jeffrey;
Disperse Viscosity, A. F. Martin;
Extractives, Reavis C. Sproull;
Lignin, F. E. Brauns;
Moisture, A. G. Scroggie;
Molecular Chain Length, H. Mark;
Pentosans, William K. Wilson;
Sampling, W. L. Stafford; and
Soda Solubles, Charles H. Lindsley.

A proposed tentative method for the determination of moisture in cellulose is being promulgated as the initial activity of the subcommittee.

Subcommittee III on Organic Esters (Leo B. Genung, chairman) is organized into three task groups as follows: A, *New Viscosity Method;* B, *Cellulose Acetate*; and C, *Mixed Esters.*

Within this subcommittee promulgation has begun on a proposed tentative method for viscosities of cellulose derivatives by the ball-drop method and a suggested revision of the Standard Methods of Testing Cellulose Acetate (D 871 - 48).

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman).—During the organizational period of the committee, W. A. Kirklin served as chairman of this subcommittee. Relatively little developmental work is anticipated since ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products has methods for cellulose nitrate.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

Subcommittee V on Cellulose Ethers (R. S. Apple, chairman) is organized into three Sections:

1. *Ethyl Cellulose* (Robert Eyler, chairman).—Existing ASTM methods for ethyl cellulose are expected to minimize developmental work in this section.

2. *Methyl Cellulose* (R. W. Swinehart, chairman).—Methods available for ethyl cellulose can be adapted for methyl cellulose.

3. *Carboxymethylcellulose* (F. W. Smith, chairman).—The program of this section comprises the development of methods for etherification, purity, and viscosity.

Subcommittee VI on Statistical Design and Analysis (E. J. Delate, chairman).—Since its primary purpose is to assist in the work of all of the subcommittees, its membership will include a representative of each of the other subcommittees.

This report has been submitted to letter ballot of the committee, which consists of 49 voting members; 38 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER,
Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing carried on activities on a number of projects through its various subcommittees and task groups. During ASTM Committee Week in Detroit, Mich., on March 2–6, 1953, meetings were held of twelve E-1 subcommittees and task groups. Arrangements have been made for meetings of 7 subcommittees and 5 task groups during the 1953 Annual Meeting of the Society. In addition, Subcommittees 17 on Thermometers and 26 on Filtration Materials met at the Corning Glass Center, Corning, N. Y., on October 8 and 9, 1952, and in Washington, D. C., on April 14 and 15, 1953. Also Subcommittee 21 on Metalware Laboratory Apparatus met in Washington on April 15.

The Board of Directors has reappointed Messrs. J. R. Townsend and W. H. Fulweiler for another term of three years and has appointed Messrs. A. C. Webber and R. C. McMaster for a term of three years as members of Committee E-1.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1952 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Cell Type Oven with Controlled Rates of Ventilation (E 95 – 52 T).

Revision of Tentative Method of:

Testing and Standardization of Etched-Stem Liquid-In-Glass Thermometers (E 77 – 49 T).

These recommendations were accepted by the Standards Committee on September 5, 1952, and the new and revised tentatives appear in the 1952 Book of ASTM Standards, E 95 in Part 6, and E 77 in Part 5.

On October 23, 1952, the Administrative Committee on Standards accepted the following recommendations submitted by Committee E-1:

Revision of Tentative Method for:

Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T), and Compression Testing of Metallic Materials (E 9 – 51 T), the revision comprising a combination of Methods E 9 and E 78.

Withdrawal of Tentative Method of:

Compression Testing of Metallic Materials in Sheet Form (E 78 – 51 T).

The revised methods appear in the 1952 Book of ASTM Standards; E 70 in Parts 4, 5, 6, and 7, and E 9 in Parts 1 and 2.

Revisions of the Tentative Method of Verification and Classification of Extensometers (E 83 – 50 T) were accepted by the Standards Committee on November 18, 1952. The revised method appears in Parts 1, 2, and 6 of the 1952 Book of ASTM Standards.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

NEW TENTATIVE

Tentative Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form.—The committee recommends these methods for publication as tentative as appended hereto.[1] These new methods were developed by the Task Group on Water Vapor Permeability (Robert Burns, chairman) and represent the results of approximately four years of cooperative work in correlating the various ASTM methods for water vapor transmission. They are applicable to a variety of materials such as paper, plastic film, and sheet materials. Five procedures for water permeability under different test conditions are covered. These methods were developed cooperatively with representatives of those ASTM technical committees having a major interest in the problem; in addition improvements have been made aimed at extending the usefulness of the methods for committees not having procedures of their own. It is hoped the methods will be found generally acceptable and will eventually replace the existing methods.

TENTATIVE REVISION OF STANDARD

Kinematic Viscosity Thermometers.—The committee also recommends the three new Specifications for ASTM Kinematic Viscosity Thermometers having temperature ranges of −2.5 to +2.5 F, −42.5 to −37.5 F, and −67.5 to −62.5 F, as appended hereto[2] for publication as tentative as part of the Specifications for ASTM Thermometers (E 1 - 52)[3]. These new thermometer specifications were prepared at the request of Committee D-2 on Petroleum Products and Lubricants for use in the revised ASTM Tentative Methods of Test for Kinematic Viscosity (D 445 - 53 T).[4] Since two of these thermometers will be used at temperatures below the freezing point of mercury (−40 F) they are constructed with a mercury-thallium alloy as the actuating liquid.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of the Standard Specifications for ASTM Thermometers (E 1 - 52)[3] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Table I.—Revise the various thermometers in this table as follows:

On ASTM Bomb Calorimeter Thermometer 56 F, change the standardization interval from "5 F" to "2.5 F."

Continue to specify glass ring only for top finish on thermometers 56 C and 56 F.

Change ASTM Aniline Point Thermometers 34 C and 34 F to eliminate contraction chamber. This requires slight revisions in dimensions as follows: bulb length 10 to 19 mm, bottom of bulb 106 to 120 mm, top of thermometer to graduation line 30 to 45 mm.

Change ASTM Solvents Distillation Thermometer 38 C to eliminate contraction chamber. This revision changes dimensions as follows: bottom of bulb to 24C–126 to 146 mm, top of thermometer to 78C–26 to 46 mm.

Change all Fahrenheit Thermometers to specify the immersion in millimeters rather than in inches.

Change Saybolt Viscosity Thermometers 17 C to 22 F, inclusive, to specify an arrow marking the test points in place of a contrasting color.

Change ASTM Solvents Distillation Thermometer 37 C to include 0 C as a standardization temperature.

Change all Partial Immersion Thermometers to include a tolerance of ±0.5 mm on specified location of the immersion line.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends for adoption as standard the 18 Specifications for ASTM Precision Thermometers 62C to 70C and 62F to 70F as published in

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[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 7.

[2] The new specifications were accepted by the Society and appear as part of Standard E 1 - 53 in the 1953 supplement to Book of ASTM Standards, Part 5.

[3] 1952 Book of ASTM Standards, Part 5.

[4] The revised methods appear in the 1953 Supplement to Book of ASTM Standards, Part 5.

Table I of the Standard Specifications for ASTM Thermometers (E 1 – 52)[3] and requests that this proposal be approved for reference to letter ballot of the Society.

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—This subcommittee has a task group considering the use of balances and weights for the calibration of hardness testing machines. Under consideration also is the need for a portable extensometer calibrator for testing machines since these devices are now available from several sources.

Subcommittee 2 on Effect of Speed in Mechanical Testing (P. G. McVetty, chairman).—During the past year the principal work of this subcommittee has been divided among four task groups.

The *Task Group on Bibliography* under B. L. Wilson, chairman, has brought up to date the bibliography on speed of testing, last published in 1947, by the addition of 213 references and abstracts. This material is ready for publication.

The *Task Group on Metallic Materials* under F. M. Howell, chairman, has considered the specifications of the American Iron and Steel Institute and suggestions for changes in the speed requirements of the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 52 T). As soon as the current revision of Federal Specification QQ-M-151 is available, the revision of Methods E 8 will be reconsidered.

The *Task Group on Non-Metallic Materials* under F. G. Tatnall, chairman, has work in progress on the correlation of speed requirements for various nonmetallic materials.

The *Task Group on Testing Machines and Speed Control Equipment* is being reactivated under its new chairman N. H. Murdza.

Subcommittee 3 on Elastic Strength of Materials (F. M. Howell, chairman).—The work of this subcommittee has been centered in its two task groups as follows:

The *Task Group on Elastic Constants* under Walter Ramberg, chairman, is undertaking the preparation of a tentative method for determining Young's modulus for metals at room temperature. Following completion of this a recommended practice will be developed for the determination of Poisson's ratio and shear modulus at room temperature. The need for these methods has been indicated as the result of the Symposium on Determination of Elastic Constants[5] which was sponsored by this subcommittee in June, 1952. The chairman of the task group presented to the committee an interesting summary of methods for determining elastic constants that are being used in European laboratories based on information obtained on his attendance at the Eighth International Congress for Applied Mechanics held in Istanbul in August, 1952.

The *Task Group on Correlation of Definitions Relating to Methods of Testing* under P. G. McVetty, chairman, has considered at length the problems involved in revising the Standard Definition of Terms Relating to Methods of Testing (E 6 – 36). Considerable information has been obtained as a result of three articles and discussions published in the ASTM BULLETIN[6] and from a questionnaire to members of Committee E-8 on Nomenclature and Definitions. The first proposed revision will be limited to thirteen terms. The Books of ASTM Standards have been surveyed for various ASTM definitions of these terms and compilations of existing definitions and suggestions for changes are being prepared. This background material will be used to draft the proposed revision. It is expected that this will be ready for review by Subcommittee 3 in March, 1954.

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[5] Issued as separate publication *ASTM STP No. 129.*

[6] "ASTM Definitions of Terms Relating to Methods of Testing," ASTM BULLETIN, No. 182, May, 1952, p. 66; discussion, No. 185, October, 1952, p. 25, and No. 187, January, 1953, p. 41.

Subcommittee 4 on Tension Testing (R. L. Templin, chairman).—A task group has been appointed to consider revisions of the definitions in the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 52 T) to correspond with revised definitions for the terms yield point, yield strength, reduction of area, etc., now being prepared by Subcommittee 3.

The subcommittee considered a request from Mr. W. R. Willets, representative of Committee D-6 on Paper and Paper Products, that a task group be appointed to undertake preparation of methods for tension testing of non-metallic sheet materials. It was also suggested that the subcommittee should sponsor a symposium on tension testing of non-metallic materials in order to obtain data on processes and techniques now being followed in the testing of various types of non-metals. If sufficient interest is evidenced in this project a task group will be appointed.

The subcommittee reviewed a proposed Tentative Method of Testing Sandwich Constructious in Tension in Flatwise Plane prepared by Committee C-19 on Structural Sandwich Constructions and submitted comments and suggestions for consideration.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—The Tentative Method of Test for Brinell Hardness of Metallic Materials (E 10 – 50 T) has been reviewed and recommendations for revision are being formulated. A test method for rapid direct-reading types of indentation hardness tests is being drafted. Portable indentation hardness testers are also under study. The procedure of applying the major load in Rockwell hardness testing is under study.

A Survey of Investigations of Effect of Sample Thickness on Rockwell Tests has been prepared in cooperation with Subcommittee W-1, of Committee B-5 on Copper and Copper Alloys, and will be submitted for publication in the ASTM BULLETIN.[7]

A Correlation of Published Data for Rockwell "C" and "30 N" Corrections for Cylindrical Specimens has been prepared and this will also be offered for publication after editorial review.[8]

Subcommittee 6 participated in the meeting of Technical Committee 17 on Steel, of the International Organization for Standardization, held in New York at Columbia University on June 9 to 12, 1952.

Subcommittee 7 on Impact Testing (W. W. Werring, chairman).—This subcommittee is now voting on some proposed revisions in the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 47 T).

This subcommittee has under consideration the matter of sponsoring a symposium on impact testing in order to present up-to-date information on newer types of impact tests now being used for various materials.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman).—The Task Group on Absolute Viscosity has taken steps to organize an American group to advise on rheological matters in connection with Technical Committee 66 on Viscosity, of the International Organization for Standardization.

The Standard Definition of Terms Relating to Rheological Properties of

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[7] R. H. Heyer and V. E. Lysaght, "Survey of Investigations of Effect of Specimen Thickness on Rockwell Test," ASTM BULLETIN, No. 193, October, 1953, p. 32 (TP164).

[8] R. S. Sutton and R. H. Heyer, "Correlation of Published Data for Correction of Rockwell Diamond Penetrator Hardness Tests on Cylindrical Specimens," ASTM BULLETIN, No. 193, October, 1953, p. 40 (TP 172).

Matter (E 24 – 42) are still under review by the task group under the chairmanship of R. N. Traxler.

The Proposed Method of Test for Furol Viscosity of Asphalts at High Temperatures prepared by the task group under the chairmanship of R. R. Thurston has been approved by letter ballot. Consideration is now being given to the publication of this method as tentative through submittal to the Standards Committee.

The Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 51 T) is recommended for continuation as tentative by the task group under the chairmanship of W. A. Kirklin. The task group is awaiting recommendations from the Subcommittee on Softening Point, of Committee D-4 on Road Materials, regarding revised instructions for the preparation of asphalt samples. Also additional collaborative tests using the powder method for preparation of resin samples are under way in Subcommittee XI on Resins, of Committee D-1 on Paint Materials.

Subcommittee 11 on Subsieve Testing (L. T. Work, chairman).—This subcommittee has the dual responsibility for watching the trends in the field, and for taking specific steps when these are indicated. Observations made during the past year have indicated no pressing responsibilities in the direction of revising the existing methods. Plans for a general review of the field are being considered as a future activity through sponsorship of a symposium on particle size measurement.

Subcommittee 12 on Methods for Density (Wilmer Souder, chairman).—This subcommittee has given consideration to the need for a general method or methods of test for density. As a first step it was agreed that the various methods for determining density now published by the Society should be classified and reviewed. A task group under the chairmanship of J. J. Moran was accordingly appointed to proceed with this project.

Subcommittee 14 on Conditioning and Weathering (A. C. Webber, chairman).—A list of essential requirements for enclosures for high humidity testing was prepared by this subcommittee at its March meeting. These proposed requirements have been referred to a task group as a basis for preparing a proposed standard. The subcommittee has also decided to undertake the preparation of general methods of determining relative humidity, and especially humidities in small enclosures, since the need for a more up-to-date method than the present ASTM Standard Method of Determining Relative Humidity (D 337 – 34) has been indicated.

Subcommittee 15 on Chemical Analysis (J. W. Stillman, chairman).—The first draft of Proposed Recommendations on Form of ASTM Methods for Chemical Analysis has been completed by a task group consisting of W. A. Kirklin, C. K. Rice, and J. W. Stillman following three meetings held at ASTM Headquarters. This draft will be presented to the subcommittee at its June meeting.

The subcommittee is still exploring the possibility of a publication containing the ASTM Methods for the Chemical Analysis of Non-Metallic Materials. The existing methods for analysis of the inorganic non-metallic materials seem to hold the most promise for such a consolidation.

Subcommittee 17 on Thermometers (R. M. Wilhelm, chairman).—This subcommittee has been especially active as indicated by the several new specifications for thermometers and recommended changes in the existing Specifications for ASTM Thermometers presented earlier in this report.

Subcommittee 18 on Hydrometers (R. D.

Thompson, chairman).—During the past year the subcommittee continued its work on the preparation of specifications for hydrometers. A milestone was reached in the publication of the Proposed Specifications for ASTM Hydrometers in the appendix to the 1952 Report of Committee E-1.[9] Included in these specifications are the 30 API hydrometers, and in addition, 10 API Thermo-Hydrometers having the thermometer scale in the body along with 4 API Thermo-Hydrometers having the thermometer scale in the stem. Two new soil hydrometers are also included.

The Task Group on Specifications plans to continue work in this field, the next step being the preparation of specifications for specific gravity hydrometers for both light and heavy liquids.

A task group was appointed in 1952 to develop methods of testing hydrometers.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman).—The subcommittee has continued to serve in an advisory and consulting capacity to the technical committees of the Society on problems dealing with laboratory glassware. An important accomplishment during the year was the completion of specifications for a new distilling flask which will replace the 100-ml Engler flask used in various distillation tests by a number of committees.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman).—The Specifications for Cell Type Oven with Controlled Rates of Ventilation (E 95 – 52 T) have now been published as tentative by the Society. Arrangements have been made by the subcommittee to display at the Annual Meeting in June laboratory ovens made in accordance with these specifications. It is expected that this exhibit will be of special interest to a number of technical committees.

The subcommittee has given consideration during the year to the following types of metalware apparatus: grease testing equipment, Engler viscosimeters, flash point testers, ring-and-ball softening point apparatus.

Subcommittee 22 on Hydrogen Ion Determination (E. B. Ashcraft, chairman).—The Revised Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 52 T) was accepted by the Society in October, 1952. The first draft of a Tentative Method for the Colorimetric Determination of the pH of Aqueous Solutions has been prepared and circulated within the subcommittee for comment. Some of the suggested changes are rather extensive and it is not anticipated that agreement on the method can be reached before next year.

Subcommittee 26 on Filtration Materials (T. L. King, chairman).—This subcommittee has been engaged in a study of the properties of rigid porous filters during the past two years. The problems have been defined and substantial agreement reached as to the properties to be measured and the test methods to be used. Work is now progressing toward early completion of proposals for test methods and specifications to be submitted to the committee.

Task Group A on Distillation Tests (J. S. Fawcett, chairman).—This task group under its new chairman has been continuing its review of the various ASTM distillation tests. It has also been cooperating with the Joint D-1–D-2 Committee on Distillation Tests in a review of the proposed scopes of the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 88 – 52) and Tentative Method of Test for Distillation Range of Lacquer Solvents aud Diluents (D 1078 – 52 T).

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[9] See Editorial Note, p. 495

Task Group B on Low-Temperature Testing of Elastomers and Plastics (R. S. Havenhill, chairman).—During the year 1952, Task Group B was expanded to include three active sections on low-temperature tests.

Section I on Stiffness Tests (F. M. Gavan, chairman) presented the final report on its work. This comprehensive report of over 50 pages contained not only the graphic results of nine stiffness tests on crude rubber, GR-S, and polyethylene at temperatures ranging from +73.4 F to −80 F, but also pertinent comments on the limitations and advantages of this work which was carried out by eleven different laboratories. It was decided to publish in the technical journals a condensed version of this report. This condensed report will be prepared by F. S. Conant who will cooperate with F. M. Gavan, W. E. Scoville, L. Boor, and R. S. Havenhill.

As a result of this work, recommendations for certain changes in the Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746 - 52 T) will probably be made by Committee D-20 on Plastics. Plans are under way by this subcommittee for consideration of brittleness tests and tests for rheological properties.

Section II on Hardness Round-Robin Tests (W. E. Scoville, chairman) has prepared a detailed analysis of the eight hardness instruments used. The results of tests obtained by nine laboratories on natural rubber, GR-S, non-rigid vinyl, and polyethylene at temperatures from +73.4 F to −80 F have been received and will be correlated by Ross Shearer.

Section III on Correlation of Bureau of Ships Tests (J. R. Britt, replacing C. F. Greenleaf as chairman) has reported a correlation between tests run with several different hardness testers which may possibly be close enough for specification work. A detailed report is under preparation.

Task Group C on Water Vapor Permeability (Robert Burns, chairman).—This subcommittee completed the Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form which are being submitted as tentative as mentioned earlier in this report.[1]

This report has been submitted to letter ballot of the committee, which consists of 49 voting members; 21 members returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

ASTM Hydrometers (E 100 - 53 T).

Tentative Revision of Standard Specifications for:

ASTM Thermometers (E 1 - 53).

These recommendations were accepted by the Standards Committee on September 9, 1953, and Tentative Specifications E 100 appear in Parts 3 and 5 of the 1953 Supplement to Book of ASTM Standards, and the tentative revision of Standard Specifications E 1 appears in Parts 3, 4, and 5 of the 1953 Supplement.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year, the first in New York, N. Y., on June 25, 1952, and the second in Pittsburgh, Pa., on March 6, 1953. Meetings of the Advisory Committee were held in New York, N. Y., on June 25, 1952; in Boston, Mass., on October 9, 1952; and in Pittsburgh, Pa. on March 5, 1953.

The committee has submitted the following material to the Society for publication:

(*1*) Chemical Analysis of Inorganic Solids by Means of the Mass Spectrometer. This collection of three papers and discussion, representing the symposium held at the 1951 Annual Meeting, is being published by the Society as Special Technical Publication *STP No. 149*.

(*2*) Methods for Emission Spectrochemical Analysis.[1] This will be a book containing 53 suggested methods, 3 suggested practices, a suggested nomenclature in applied spectroscopy, and several tentative methods. This publication will be the first extensive compilation of spectrochemical methods.

(*3*) Index to the Literature on Spectrochemical Analysis, Part III, 1946–1950, by B. F. Scribner and W. F. Meggers.[2] This is an extension of a bibliography which in earlier editions covered the literature from 1920 to 1945.

Two proposed new tentative methods have been prepared for action of the Administrative Committee on Standards. The first is a revision of former Tentative Methods E 26 and E 27, in which the methods are combined under the title of Tentative Method for Spectrochemical Analysis of Zinc-Base Alloys and Zinc by the Solution – D-C Arc Technique. The second is a proposed new tentative method entitled Tentative Method for Spectrochemical Analysis of Aluminum and its Alloys by the Point-to-Plane Spark Technique.

The committee, through its vice-chairman, E. B. Ashcraft, has arranged for a symposium on Fluorescent X-ray Spectrographic Analysis for the 1953 Annual Meeting.[3]

Subcommittee Activities

Subcommittee I on Apparatus and Equipment (R. G. Russell, chairman).—A revised draft of a report on Description and Specification of Spectrographic Equipment is being prepared. Descriptions of spectrographic excitation sources and other apparatus, and methods for testing equipment, also are being prepared. A procedure for testing experimental emulsions has been prepared by A. J. Mitteldorf in conjunction with the Eastman Kodak Co. Three photographic emulsions are included in the present program.

Subcommittee II on Fundamental Methods (R. W. Smith, chairman).—Group 1 on Specification of Spark Generators, headed by R. A. Wolfe, held two meetings to discuss means of carrying out cooperative testing in the comparison of spark generators. The group is now engaged in selecting and measuring suitable line pairs to be employed in the testing. Group 2, headed by Jacob Sherman, continued the preparation of a revised version of Specifications of Precision and Accuracy. The work of Group 3 was completed, for the present, by the preparation of the Suggested Practice for Photographic Photometry, by Cyrus Feldman, which is to be included in the forthcoming book. Group 4 has completed the Suggested Practice for Photographic Processing, prepared by H. A. Tuttle; this is also to be included in the book and is being prepared for acceptance as a tentative practice. Group 5 on Basic Calculations of Spectrographic Analysis has circulated a report by J. H. Coulliette and M. F. Hasler, within Subcommittee II. Group 6 on Semiquantitative Methods, headed by D. L. Fry, is preparing five types of samples with different matrices; these will be circulated for the testing of semiquantitative methods of analysis.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Published October, 1953.

[2] Issued as separate publication *ASTM STP No. 41-C*.

[3] Issued as separate publication *ASTM STP No. 157*

Subcommittee III, Editorial (C. L. Guettel, chairman) has completed the reviewing and editing of the material for the forthcoming book of suggested methods. V. A. Fassel has been appointed chairman of the Nomenclature Group; this group represents Committee E-2 on the Joint Committee on Nomenclature in Applied Spectroscopy. Cyrus Feldman has been appointed chairman of the Translation Service group, which plans considerable activity in this field.

Subcommittee IV on Standard Samples and Pure Materials (G. A. Nahstoll, chairman) is actively engaged in the study of graphite electrodes, particularly with respect to methods of testing for purity and the adoption of standard shapes and sizes of preformed electrodes. This group activity is headed by W. J. Edgar.

Subcommittee V on Copper, Nickel, and Their Alloys (E. K. Jaycox, chairman) is engaged in establishing methods for sampling copper-base alloys, through a group headed by W. A. Young. The Suggested Method for the Spectrochemical Analysis of Nickel Alloys by the D-C Arc Technique is being rewritten for conversion to tentative status.

Subcommittee VI on Lead, Tin, Antimony, Bismuth, and Their Alloys (E. J. Dunn, Jr., chairman) represented Committee E-2 on the Panel Discussion on Analysis of Tin, a part of the Symposium on Tin held at the 1952 Annual Meeting of the Society (published as Special Technical Publication No. 141 (1953)). A suggested method for the analysis of antimony metal is being revised prior to distribution to Committee E-2 members. Tentative methods are being prepared for the analysis of tin and of lead.

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (J. R. Churchill, chairman) prepared the proposed Tentative Method for the Spectrochemical Analysis of Aluminum and its Alloys by the Point-to-Plane Spark Technique.

Subcommittee VIII on Zinc, Cadmium, and Their Alloys (Alan Goldblatt, chairman) prepared the proposed Tentative Method for the Analysis of Zinc-Base Alloys and Zinc by the Solution - D-C Arc Technique. A second draft has been prepared of a tentative method for the analysis of zinc alloys using the point-to-plane and cast-pin spark techniques.

Subcommittee IX on Ferrous Metals (P. R. Irish, chairman) has been engaged in the preparation of a tentative method for the spark spectrochemical analysis of steel and cast iron. A second draft of the method is under review. A group on Sampling of Ferrous Metals, headed by E. R. Vance, has prepared a procedure which has been reviewed by the subcommittee.

Subcommittee X on Non-Metals (W. J. Poehlman, chairman) is continuing the collection of suggested methods. A total of 19 methods has been collected, of which 12 will appear in the forthcoming book. The preparation of tentative methods is planned for alkali solutions, ores and ceramic materials, and similar substances.

This report has been submitted to letter ballot of the committee, which consists of 120 members; 105 members returned their ballots, of whom 101 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. F. SCRIBNER,
Chairman.

J. H. COULLIETTE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Spectrochemical Analysis of Aluminum and its Alloys by the Point-to-Plane Spark Technique (E 101 - 53 T).

Revision of Tentative Method of:

Spectrochemical Analysis of Zinc-Alloy Die Castings for Minor Constituents and Impurities (E 27 - 43 T), with a change in title to read "Tentative Method for Spectrochemical Analysis of Zinc-Base Alloys and High Grade Zinc by the Solution - D-C Arc Technique."

Withdrawal of Tentative Method of:

Spectrochemical Analysis of Zinc for Lead, Iron, and Cadmium (E 26 - 43 T) (Incorporated in E 27 - 53 T).

These recommendations were accepted by the Standards Committee on September 9, 1953, and the new and revised tentative methods appear in the Compilation of Methods for Emission Spectrochemical Analysis.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held its meeting at the Annual Meeting of the Society on June 23, 1952, at New York, N. Y.

At the present time the committee consists of 116 members.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Recommended Practices for Apparatus for Chemical Analysis of Metals (E 50 - 48 T)[1] be approved for reference to letter ballot of the Society for adoption as standard.

ACTIVITIES OF SUBCOMMITTEES

Editorial Subcommittee.—Two meetings of the subcommittee were held during the year. The major considerations have been (*1*) the incorporation of common procedures in methods, and (*2*) the preparation of a style manual covering the form of ASTM Methods for Chemical Analysis of Metals. In the first instance, progress has been made to the point of including a common procedure for at least one element. A special numbering system is to be devised and initially the common procedure will be repeated with each method covering the element, in order to facilitate transition to the new form of the subsequent methods book. A draft (fourth) covering the Recommendations on Form is to be written. Considerable effort has been given to defining the subject headings properly, so that no conflict with other technical committee groups will occur.

Division F on Ferrous Metals.—At the annual meeting the progress of the various task force groups was reviewed. The projects assigned these task force groups cover methods for the analysis of ferroboron and ferrotitanium, the determination of silicon in ferrotungsten, aluminum in stainless-type steels, manganese in steels (photometric), magnesium in nodular iron, columbium and tantalum in stainless-type steels, cobalt in high-alloy steels, sulfur (combustion) in plain carbon steels and cast iron, and zirconium in alloy steels.

The study of the volumetric method for silicon in ferrotungsten has been completed, as well as methods for the analysis of ferrotitanium. The first drafts of these procedures have been sent to the Editorial Subcommittee for review.

Considerable difficulty was encountered in the development work on methods for the analysis of ferroboron, which in part appeared to be due to lack of homogeneity in the standard samples used. The task group is attempting to utilize one of the ion exchange resins for the separation and determination of boron in this ferroalloy.

The work on the development of an

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] 1950 Book of ASTM Methods on Chemical Analysis of Metals.

accurate method for residual amounts of aluminum in stainless-type steels has been very difficult, and to date no entirely satisfactory procedure has been forthcoming. This subgroup has done a considerable amount of work, but as yet no definite decision has been reached as to which of the many methods studied should be recommended as a standard ASTM procedure.

Studies on a photometric method for manganese in steels have been very encouraging, and it is anticipated that a proposed procedure will be ready for editorial review in the near future.

Work on the development of a method for magnesium in nodular iron is still in progress, and a final procedure is expected sometime during the coming year.

The task group responsible for studies on the combustion method for sulfur applicable to carbon steels and cast iron is attempting to develop a method using theoretical factors.

Interesting reports are being received regarding the high-frequency induction combustion as applied to the determination of sulfur, and a portion of the task force group is investigating this approach. It was the consensus of the division that the method using empirical factors, which is in quite common use in many steel plant laboratories, should be considered as an expedient measure and accepted provisionally as a tentative ASTM method.

A large amount of work has been carried on by the task group responsible for methods for columbium and tantalum in stainless-type steels. It is anticipated that a satisfactory photometric method for these two elements will be forthcoming in the next few months.

It is anticipated that a potentiometric method for cobalt in high-alloy steels will be available for study by this task force in the next month or so.

Very little progress has been made by the task force handling methods for zirconium in tungsten steels, but an attempt will be made to get some active work under way during the coming year.

The new task force group organized for the development of standard methods for the determination of small or residual amounts of elements, such as aluminum, chromium, nickel, cobalt, etc., in high-silicon steels has been very active. The new high-silicon Bureau of Standards' sample 125a is being used in the work of this task group.

At the annual meeting of the division in 1952, it was the concensus that more attention should be given rapid photometric methods for determining small or residual amounts of various elements. In most instances these procedures utilize color reactions, which are specific for the elements being determined and are much less time consuming and are as accurate, if not more so, than the more time-consuming volumetric and gravimetric methods used heretofore. In order for the division to be progressive, more consideration will have to be given to the photometric approach, even though such methods do sometimes call for instruments that are not available in many laboratories. It is felt if such methods are adopted as standard by ASTM, laboratories called on to use the methods will equip themselves to do so.

Division S on Sampling.—Subcommittee S-2 wrote and distributed for comment a method for the Sampling of Non-Ferrous Metals and Alloys in Cast and Wrought Form for the Determination of Chemical Composition, a combination of Methods E 55 and E 88.

Work on Method E 88, in cooperation with Committee B-2, is being carried on and a task group was appointed to review the comments on it.

Another task group is starting work on sampling methods for titanium metal and alloys in cooperation with Division M.

The problem of sampling pig tin has been discussed, but since no specific tin specification has yet been issued by the Society, an attempt to write a sampling procedure was deferred until such time as a specification is written.

It was agreed upon by the committee to continue existing tentative sampling specifications as tentative.

Division N on Non-Ferrous Metals:

Subcommittee N-1 on Copper and Its Alloys has prepared methods for the analysis of beryllium-copper alloys, which should be submitted for ballot in the near future.

Methods for the analysis of fire-refined copper are progressing. The method for arsenic is ready for ballot, and cooperative work is being done on a recently submitted rhodamine-B photometric method for antimony. Some cooperative work has been submitted for selenium and tellurium. It is expected to complete this work for the next edition of the Book of ASTM Methods for Chemical Analysis of Metals.

Subcommittee N-3 on Aluminum and its Alloys.—Of the three methods under consideration (magnesium, silicon, and zinc) the work on the photometric method for silicon by the molybdisilicic acid method has been completed. This procedure shows particular advantages in the lower range of silicon as to accuracy. For aluminum-silicon alloys it compares favorably with the gravimetric SiO_2 method, with the additional advantage of a considerable saving of time.

For the determination of zinc some progress has been made applying the dithizone method.

A new approach is being tried for the determination of magnesium by titrimetric means using disodium ethylenediamine tetraacetate. The results obtained so far are most encouraging.

Subcommittee N-4 on Zinc and its Alloys has under consideration the photometric method for aluminum in zinc spelter, using eriochromecyanin R.

Additional data are being obtained before submitting the cupric-bromide and the dithizone lead methods for ballot.

Subcommittee N-5 on Precious Metals and Their Alloys reports that cooperative work is being carried out on samples of silver solder. This work has in mind the possible revision of certain analytical methods, with special reference to the determination of cadmium. Progress accomplished on special samples and synthetic solutions was discussed at a meeting held April 8. Additional approaches are to be investigated for the determination of cadmium in silver solders.

Consideration is also being given to the inclusion of several photometric methods for the determination of low percentage elements present in these alloys.

Subcommittee N-6 on Nickel and Its Alloys.—In order that the group of methods published by Committee B-4 on Electrical Resistance Alloys as proposed methods for the analysis of electronic nickel be processed through Committee E-3, a task force was set up to take care of the cooperative testing. Since the methods needed editorial treatment, it was decided, in conference with a group from Committee B-4, that some revisions be made prior to the rewriting of the methods. Three meetings of the joint group have resulted in a set of revised procedures for copper, iron, manganese, titanium, and cobalt. In addition, Committee B-4 has made available to the joint task group four samples of various alloys used in the industry in order to test these procedures. It is to be hoped that sufficient data will be on hand to enable a ballot by early fall.

Work is also progressing on the analysis for other elements: aluminum, silicon, magnesium, and sulfur in particular, and it is hoped that a group of methods will result that will be compatible with the sample-size restrictions.

Subcommittee N-9 on Magnesium and Its Alloys.—Collaborative work previously done on the procedure entitled "Copper by the Hydrobromic-Phosphoric Acid Method" has led to a revision which is currently being circulated for balloting by the subcommittee.

Adoption of specifications for zirconium in certain magnesium-base alloys have created a need for a zirconium method. Three procedures have previously been subjected to collaborative work, and a revision on one of them, the phosphate method, is being circulated for subcommittee balloting.

Division G on General Analytical Methods:

Subcommittee G-1 on Reagents has conferred with Committee D-19 on Industrial Water concerning the preference of Committee E-3 for including reagent referee grade water in the particular set of specifications in which it is to be used rather than as a part of a general specification for water. Agreement with Committee D-19 on this basis was reached.

Subcommittee G-2 on Apparatus has unanimously approved the proposal to recommend adoption of Method E 50 – 48 T as standard. Work is proceeding on the development of performance standards for analytical balances, and some exploratory work has been done toward performance standards for photometers and for electrolytic apparatus.

Subcommittee G-3 on Common Procedures is making progress toward the development of a common procedure for nickel. Information covering a preliminary study of the usual procedure for nickel in steel was received from Division F and it has been agreed to conduct a further study and to attempt to make modifications which appear to be necessary at levels of nickel in steel below 0.05 per cent. As it has been indicated that Division N plans to replace the gravimetric nickel method with photometric methods, it would appear that this division will have no further interest in the common procedure for nickel. Members of Subcommittee G-3 have met with representatives of Divisions F and N to discuss certain discrepancies in the periodate method for the determination of manganese in various alloys.

Division M on Miscellaneous Metals.—Work on methods for the analysis of titanium is under way in cooperation with the National Advisory Committee on Titanium, Panel on Chemical Analysis. A program under the direction of the Watertown Arsenal is at present leading to methods for nitrogen, chromium, carbon, vanadium, and manganese. The interchange of information between ASTM and this group is proceeding well.

This report has been submitted to letter ballot of the committee, which consists of 116 members; 83 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. STILLMAN,
Chairman.

H. KIRTCHIK,
Secretary.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 has met twice during the year: at the Spring and Annual Meetings of the Society. Its membership is on a stable basis, and presently consists of 101 members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Preparation of Samples (F. C. Hull, chairman) is actively concerned with a major revision of Methods E 3 – 46 T on the Preparation of Samples. The newer methods and materials for sample polishing, new etchants, and many new metals and alloys that require consideration make this revision a major task. The present method of attack is by means of task-groups assigned to particular metals and their alloys.

The subcommittee is recommending the retention of Methods E 3 – 46 T in its present form, pending the above revision.

Subcommittee II on Definitions (R. S. Busk, chairman) anticipates having completed, by the end of the Annual Meeting, the task of selecting and defining the terms pertinent to the activities of Committee E-4 in the fields of microscopy, X-ray diffraction, metallography, physical metallurgy, electron microscopy, etc. Nearly a thousand terms and definitions have been considered and approved.

Subcommittee III on Nomenclature (P. A. Beck, chairman) reports material and satisfactory progress in its attempt to establish a system of phase nomenclature for metallic systems. This international group has what is hoped to be the final draft of its proposal being given a preview by 80 prominent metallurgists here and abroad. The proposal will be publicized later this year in order to solicit further comments prior to submission for formal Society approval.

Subcommittee IV on Photography (S. W. Poole, chairman) is preparing for the next photomicrographic exhibit by sponsoring its student activity phase of the program in schools and colleges here and abroad.

The Subcommittee is recommending the retention of Methods E 2 – 49 T without change.

Subcommittee V (Alexander Gobus, chairman) is actively engaged in continuation of its round-robin tests on a specially prepared group of test blocks in order to correlate inter-laboratory tests.

Subcommittee VI on X-ray Diffraction (W. L. Fink, chairman) is recommending the retention with some changes of Method E 81 – 49 T on Pole-Figure Preparation. Method E 82 – 49 T on Orientation will be re-established with a few improvements.

Due to the improvements in equipment and techniques, Recommended Practice E 43 – 49 T on Chemical Analysis is being completely revised, and will be expanded to include the most improved techniques for precision determinations. It will not be possible to conclude this work by the time of the Annual Meeting, as it is planned to use this new proposal as a basis for international cooperation in this field, most particularly as it affects the Joint Committee on Chemical Analysis by Powder Diffraction Methods and its Card Index. For this reason, it is hoped that the best practices here and abroad can be incorporated into the revised proposal, giving it international stature.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29-July 3, 1953.

Subcommittee VII on Thermal Analysis (D. I. Finch, chairman) is recommending the re-establishment without change of Recommended Practices E 14 - 51 T on Thermal Analysis and E 80 - 49 T on Dilatometric Analysis. Plans have also been made to prepare a recommended practice for thermal analysis by means of electrical resistance measurements.

Subcommittee VIII on Grain-size (R. E. Penrod, chairman) and *VIIIc* (H. P. George, chairman) are currently reviewing the differences which seem to exist in the several grain size standards, in order to unify any discrepancies. Currently, VIIIc is recommending the re-establishment without change of Methods E 79 - 49 T on Grain Size of Copper and Copper Products, and Method E 91 - 51 T on Non-Ferrous Grain Sizes (except copper, etc.)

Subcommittee XI on Electron Microscopy (W. L. Grube, chairman) is making further progress as is witnessed by the third report on their researches which, as the subcommittee report, is appended to this report.[1]

In addition, the techniques which have been proven by these investigations form the basis of the two-session symposium on these methods which the subcommittee is sponsoring at the Annual Meeting of the Society.[2]

It is anticipated that these activities will result in information which can form the basis of recommendations for recommended practices as applied to the electron microscopy of steels.

This report has been submitted to letter ballot of the Committee, which consists of 101 members; 94 members returned their ballots, of whom 87 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

M. R. NORTON,
Secretary.

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[1] See p. 505.

[2] Issued as separate publication *ASTM STP No. 155.*

APPENDIX

TECHNIQUES FOR FERROUS ELECTRON METALLOGRAPHY

Third Progress Report of Subcommittee XI on Electron Metallography*

The first two reports [1, 2] of Subcommittee XI of Committee E-4 on Metallography presented the results of a cooperative study with the electron microscope of the structures of a series of specimens of eutectoid steel prepared specially for the purpose. The structures studied to date include those resulting from the direct transformation of austenite at various temperatures, such as pearlites and bainites, and those resulting from tempering martensite, also at various temperatures.

The purpose of this work was twofold: to gain fundamental knowledge of the structures of steel and, in the process of acquiring this information, to develop techniques of electron metallography that would preclude errors of interpretation resulting from faulty preparation of the specimen or from inadequate replication methods. Specifically, the aim of the subcommittee was to examine closely every step involved in the preparation of an electron micrograph and, by a process of trial and elimination, to arrive eventually at techniques which would be generally accepted because of their soundness, reproducibility, and interpretability.

Since the structures of carbon steel of greatest metallurgical interest not entirely resolvable with the light microscope are aggregates of carbide and ferrite, such as pearlite, bainite, and tempered martensite, the work of the subcommittee has been directed toward effecting complete resolution and identification of the constituents of these structures. This has meant studying primarily the configuration and distribution of carbide particles in a ferritic matrix, although whenever structures such as retained austenite or untempered martensite have been present, they too have been examined closely.

In order to study the size and distribution of the carbides in a steel microstructure with the electron microscope, it is first necessary to prepare the surface of the specimen by polishing, and then to develop differences in relief between the carbide particles and the ferrite matrix by selective etching. A replica is prepared embodying the relief differences of the surface, and electron micrographs are taken of small regions of the replica. The smallest significant detail of the microstructure that can be observed is limited by the resolution of the etchant and by the resolution of the replica, since the microscope is capable of resolving smaller detail than either of these two techniques. This approach automatically prescribes light etching with reagents endowed with high sensitivity for carbide-ferrite differentiation. Several were investigated at the beginning of the cooperative program (Spring, 1948), and a saturated solution of picric acid in ethyl aclohol was found to be best suited for the purpose. At the same time, a trial of available replica techniques indicated that shadowcast plastic replicas would prove most useful for subcommittee use.

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* Members of Subcommittee XI actively participating in this study:

W. L. Grube, *Chairman*—General Motors Corp., Detroit, Mich.
C. M. Schwartz, *Secretary*—Battelle Memorial Institute, Columbus, Ohio
L. B. Birks, Naval Research Laboratory, Washington, D. C.
R. D. Chapman, Chrysler Corp., Detroit, Mich.
P. Coheur, Centre Belge de Metallurgie Physique, Liege, Belgium
A. L. Ellis, International Harvester Co., Chicago, Ill.
A. Feduska, Heppenstall Steel Co., Pittsburgh, Pa.
R. M. Fisher, U. S. Steel Corp., Kearny, N. J.
E. F. Fullam, General Electric Co., Schenectady, N. Y.
A. H. Geisler, General Electric Co., Schenectady, N. Y.
D. J. Girardi, The Timken Roller Bearing Co., Canton, Ohio
L. Habraken, Centre Belge de Metallurgie Physique, Liege, Belgium
J. M. Hodge, U. S. Steel Corp., Pittsburgh, Pa.
L. D. Pellier, American Cyanamid Co., Stamford, Conn.
G. E. Pellissier, U. S. Steel Corp., Pittsburgh, Pa.
G. H. Robinson, G. M. Technical Center, Warren, Mich.
F. G. Rowe, American Cyanamid Co., Stamford, Conn.
D. M. Teague, Chrysler Corp., Detroit, Mich.
C. F. Tufts, Sylvania Electric Products, Inc., Bayside, L. I., N. Y.
J. R. Vilella, U. S. Steel Corp., Kearny, N. J.

[1] "Electron Microstructure of Steel First Progress Report of Subcommittee XI on Electron Microstructure of Steel, ASTM Committee E-4 on Metallography," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 444 (1950).

[2] "Electron Microstructure of Bainite in Steel. Second Progress Report by Subcommittee XI of Committee E-4," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 543 (1952)

As a result of that preliminary work, the subcommittee standardized on shadowcast negative plastic replicas of specimens mechanically polished and etched with 4 per cent picral for studying the microstructures of eutectoid carbon steel. Recently, however, the subcommittee decided to resurvey replica and etching techniques to insure that the most suitable methods were being employed to meet the objectives of the subcommittee.

The results of the re-examination of techniques are reported here in two parts. Part I describes the work on replica techniques, and Part II discusses the results obtained by using various etching procedures on the eutectoid carbon steel microstructures.

PART I.—SILICA AND PLASTIC REPLICAS

The replica method employed by the subcommittee in its current research program must be well suited to plain carbon steels and easy to interpret in terms of the size, shape, and distribution of the carbide particles in the various microstructures. The method should also meet the following general requirements:

(*1*) It should be familiar to most electron metallographers and commonly used in many laboratories,

(*2*) It should be easy to apply to steel specimens prepared in the same manner as for light microscopy, and

(*3*) The replica technique should not harm the sample, and it should be possible to make several successive replicas of the same prepared surface.

Of the replica methods available,[3] only two are considered to meet all the above requirements, namely, the shadowed negative plastic and plastic–silica techniques. Other replica techniques, such as those recently reviewed by Heindenreich,[4] are either still in process of development and not yet in general use or are specific to metals other than plain carbon steels.

To evaluate the relative merits of the silica and plastic methods for subcommittee use, two of the specimens of the previous reports were re-examined, using both techniques after etching with an alcoholic solution of 4 per cent picral.

EXPERIMENTAL WORK

The specimens selected for intercomparison of the two replica methods were Nos. 1 and 5 discussed in the previous reports and consisted, respectively, of the coarse pearlite and lower bainite microstructures of the plain carbon steel containing 0.87 per cent carbon used by the subcommittee. The carbide particles can be adequately resolved by the light microscope in the coarse pearlite specimen, but they are irresolvable in the lower bainite specimen.

The heat treating procedures employed in preparing these specimens are listed in Table I. Further details on the two specimens, including photomicrographs at 2500 ×, and also the chemical analysis and isothermal transformation diagram of the steel used, can be found in the first two subcommittee reports.[1, 2]

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[3] A comprehensive discussion of replica methods will be found in the *Journal* of the Microscopical Society, Vol. 70, Chapter V (1950).

[4] R. D. Heindenreich, "Methods in Electrón Microscopy of Solids." *Review of Scientific Instruments*, Vol. 23, November, 1952, p. 583.

The two replica procedures, including variations from the original techniques, are described in detail in the Appendix to Part I. Briefly, they are as follows: The negative plastic technique consists of coating the polished and etched surfaces of the specimen with plastic dissolved in a solvent and then stripping this thin film from the surface after the solvent evaporates. The replica is shadowcast by oblique deposition of metal by vacuum evaporation onto the replicating surface before examination in the electron microscope. In the silica methods, the prepared surface is first replicated on a thick plastic film either by molding a thermoplastic under pressure and heat against the surface, or by evaporation of the solvent from a concentrated solution of plastic flowed over the surface. This first replica is next coated with a thin film of silica evaporated normally to the surface. After the silica film is separated from the plastic and washed, it is ready for examination in the electron microscope.

TABLE I.—HEAT TREATMENTS OF STANDARD SPECIMENS.

Sample	Structure	Heat Treatment[a] Following Austenitizing	Rockwell, Hardness C Scale
No. 1.......	Coarse pearlite	3½ hr at 1300 F	13.8
No. 5.......	Bainite	2½ hr at 500 F	57.4

[a] All these specimens were austenitized at 1800 F fo 35 min and were air-cooled from the final indicated temperature.

A variation of the silica technique is to metal shadowcast the first plastic replica prior to deposition of the silica. Several electron micrographs of replicas prepared in this way are included for comparison.

The electron micrographs of the replicas were direct printed at a final magnification of 15,000 ×, so that the shadows appear white. The micrographs shown are generally representative of the more than 75 submitted by members of the subcommittee.

RESULTS

The electron micrograph of Fig. 1 illustrates the result of using the shadowed plastic technique on the coarse pearlite specimen prepared by isothermal transformation of the eutectoid steel at 1300 F. The lamellar structure can be seen clearly and the relative relief of the carbide particles and the ferrite matrix determined from the shadows. Metal shadowing may create the illusion that the particles of the carbide phase standing in relief after etching the metal surface are smaller in the shadowing direction than they actually are, due to the fact that the shadow lies inside the carbide. However, this illusion may be minimized by shadowing at a higher angle.

Figure 2 is an electron micrograph of a molded polystyrene–silica replica of the same coarse pearlite specimen shown in Fig. 1. It demonstrates that the main contrast developed by a silica replica is along abrupt changes in surface relief, as at the phase boundaries between the slow etching carbide lamellae and the more rapidly etching ferrite. There is no difference in tone between the ferrite valleys and carbide plateaus, and the only good way to determine the relief of such a replica is by the use of stereoscopic techniques.

Occasionally a light band can be seen running parallel to the dark contour lines, as in the electron micrograph of the cast Parlodion–silica replica of Fig. 3. This is a result of silica self-shadowing

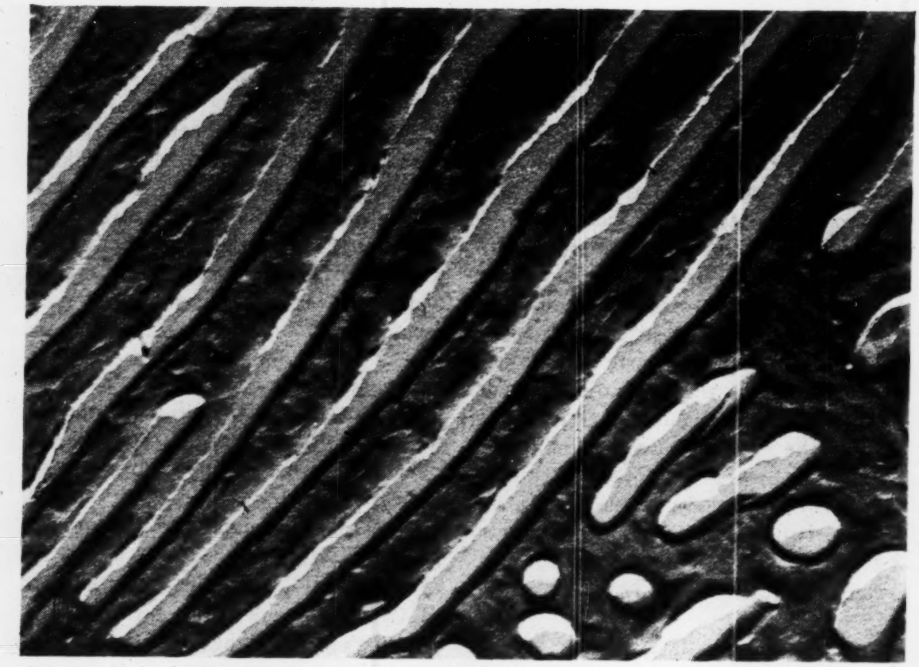

FIG. 1.—Electron Micrograph of Shadowed Collodion Replica of Coarse Pearlite, Isothermal Transformation at 1300 F (× 15,000).

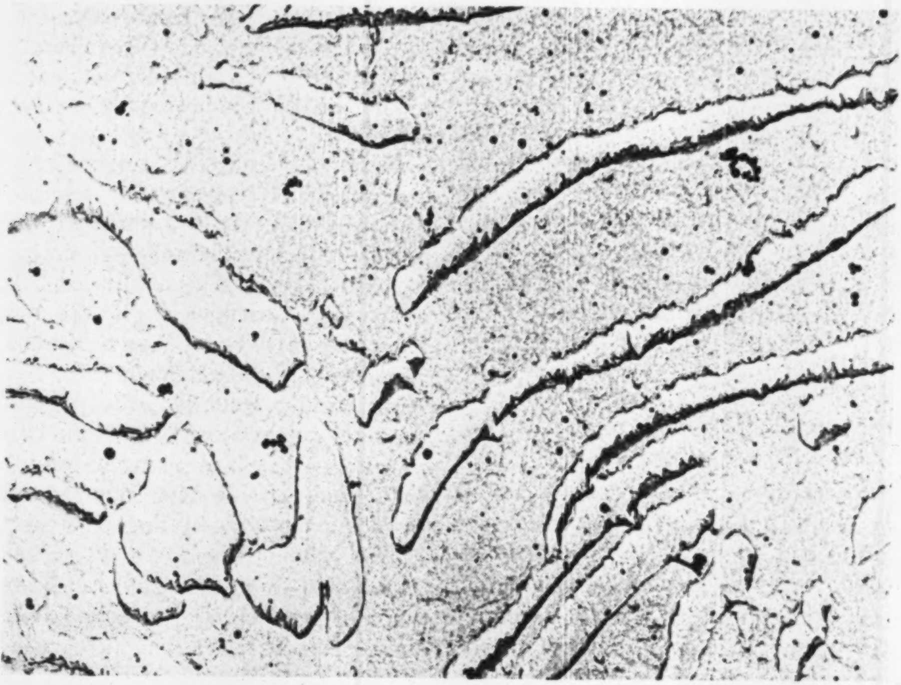

FIG. 2.—Electron Micrograph of Polystyrene–Silica Replica of Coarse Pearlite, Isothermal Transformation at 1300 F (× 15,000).

due to inadvertent oblique deposition of silica during the evaporation. The relative relief of detail on the surface may be determined from the relationship of this shadow band to the contour line.

The electron micrograph in Fig. 4 of a molded polystyrene–silica replica shows the result of shadowing the polystyrene with metal before depositing the silica film. The silica coating serves mainly as a support for the metal shadow film; this figure can be compared with the micrograph of the shadowcast negative plastic replica of Fig. 1. The micrograph of Fig. 5 shows another intermediate shadowed plastic–silica replica in which a cast Parlodion film was used for the first replica. The relative relief of the carbide and ferrite can be determined in both these figures from the metal shadows.

All of the electron micrographs of shadowed plastic replicas of this sample were of good quality, while several micrographs of molded polystyrene–silica replicas showed fine over-all granularity of the evaporated silica film, and others showed the result of strain markings that formed in the polystyrene. Both the regular and the intermediate shadowed molded polystyrene–silica replicas, as in Figs. 2 and 4, show serrated edges on the carbide particles and fine dotted structure in the ferrite. There is serious doubt as to the authenticity of either of these structures.

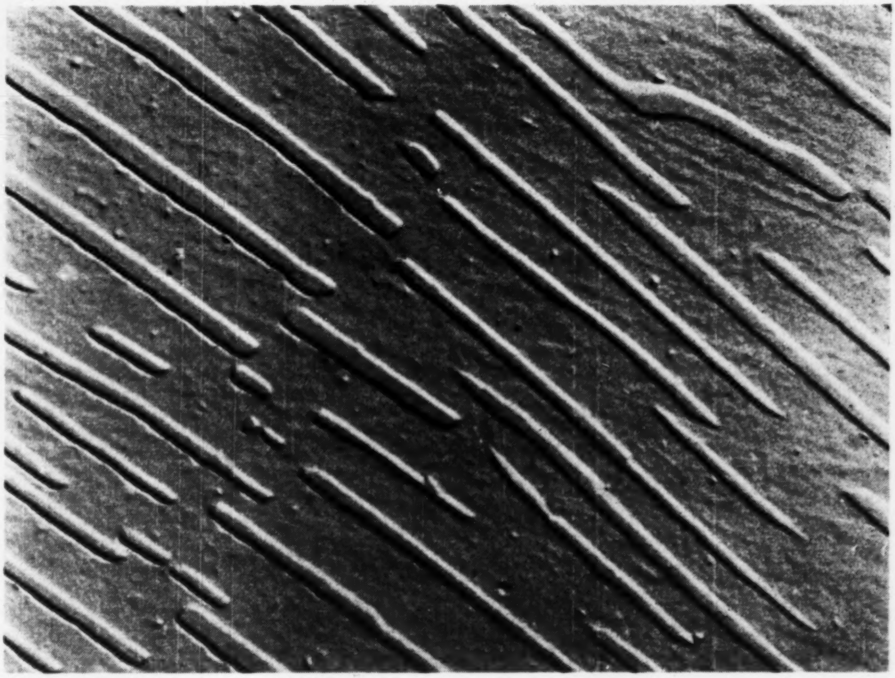

FIG. 3.—Electron Micrograph of Parlodion–Silica of Coarse Pearlite, Isothermal Transformation at 1300 F (× 15,000).

The electron micrograph of Fig. 6 shows a shadowed plastic replica of lower bainite prepared by isothermal transformation at 500 F. The parallel array of small carbide particles within the ferrite needles can clearly be seen and are shown oriented at an angle of ap-

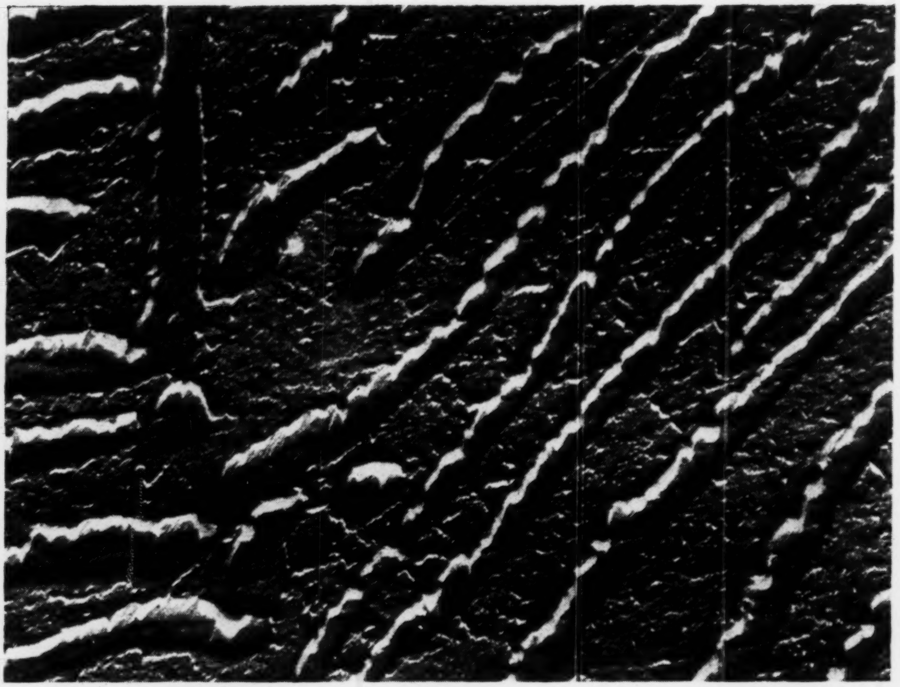

FIG. 4.—Electron Micrograph of Intermediate Shadowed Polystyrene–Silica Replica of Coarse Pearlite, Isothermal Transformation at 1300 F, Picral Etch (× 15,000).

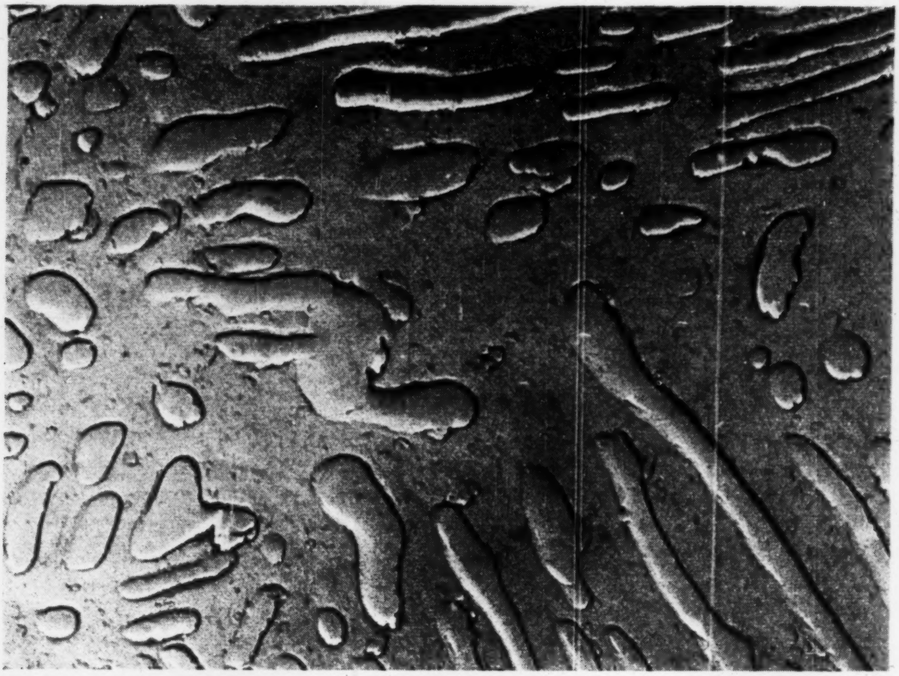

FIG. 5.—Electron Micrograph of Pre-Shadowed Parlodion–Silica Replica of Coarse Pearlite, Isothermal Transformation at 1300 F (× 15,000).

proximately 55 deg with respect to the needle axis. These carbide particles appear to be thin platelets; the apparent thickness of these platelets probably depends on the angle at which they intersect the surface. The silica replica of this same bainite sample is shown in Fig. 7. This micrograph is very difficult to interpret even when we have an understanding of the structure from the micrograph of the shadowed plastic replica. Intermediate shadowing of the cast Parlodion film before silica coating, as in Fig. 8, facilitates interpretation of the structure.

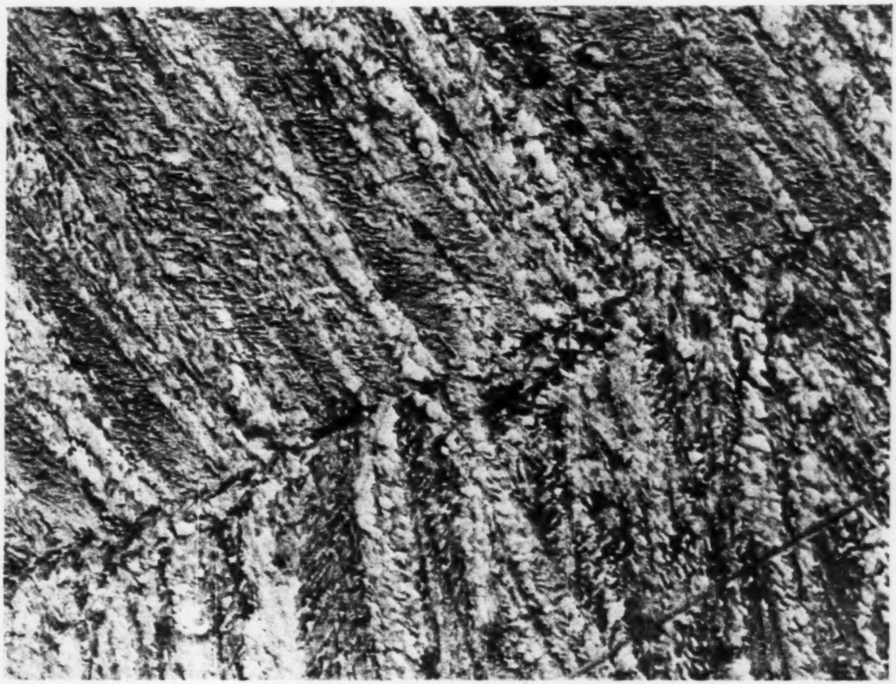

FIG. 6.—Electron Micrograph of Shadowed Collodion Replica of Lower Bainite, Isothermal Transformation at 500 F (× 15,000).

SUMMARY OF RESULTS

Comparison of the electron micrographs of the two test specimens points up the differences in results obtained by the use of plastic and silica methods. The shadowed plastic replicas are easy to interpret because of differences in tone developed between unetched carbide particles and ferrite, and because of the shadows cast by projections on the replica surface. The high contrast developed only along very sharp changes in relief, plus the lack of tone differences between carbide and ferrite, makes silica replicas very difficult to interpret.

In comparing the replicas on the basis of resolution, it must be emphasized that the resolution of a replica can be no better than the resolution of the first plastic impression. Pressure molded polystyrene might be expected to show higher resolution than impressions of cast plastics. This may be the case on some surfaces, but the silica replicas of molded polystyrene impressions of the specimens in this investigation showed considerable

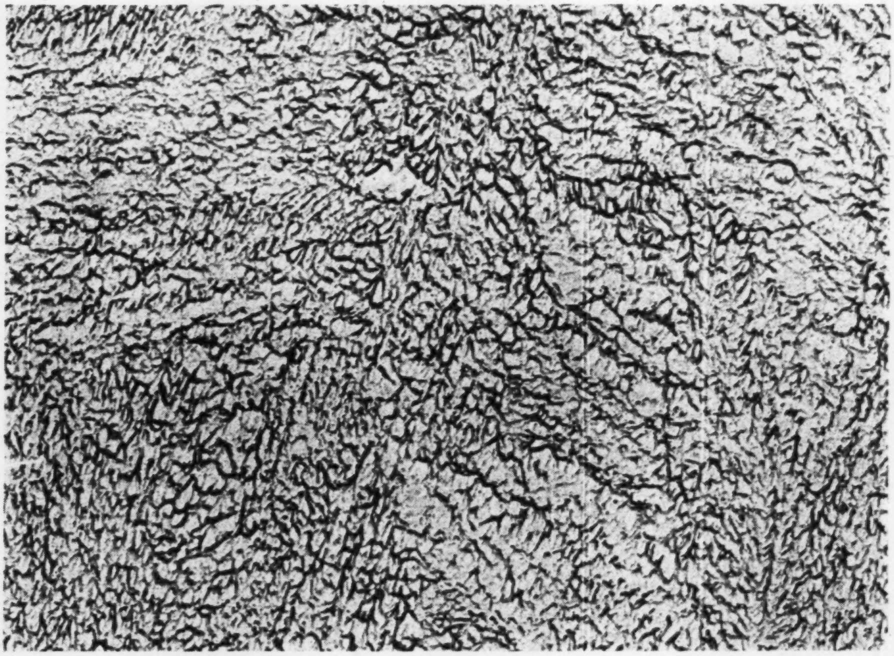

FIG. 7.—Electron Micrograph of Polystyrene-Silica Replica of Lower Bainite, Isothermal Transformation at 500 F (× 15,000).

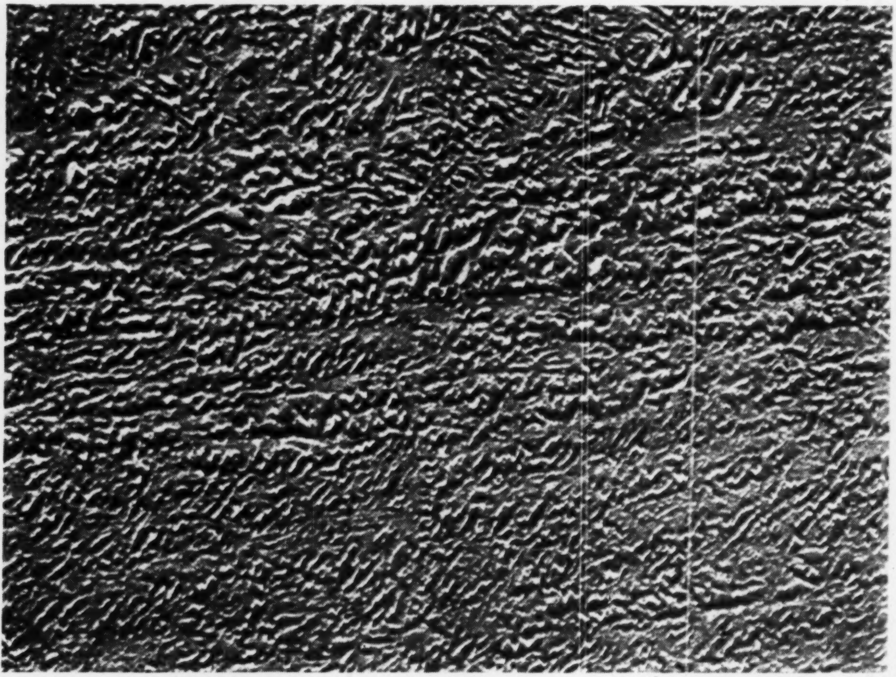

FIG. 8.—Electron Micrograph of Pre-Shadowed Parlodion-Silica Replica of Lower Bainite, Isothermal Transformation at 500 F (× 15,000).

artifact. The carbides appeared to have serrated edges, and there appeared to be a great deal of fine structure in the ferrite. These effects may be the result of plastic distortion or fracture of the polystyrene during stripping and are most pronounced in undercut regions along the edges of carbides and in etch pits. The shadowed plastic replicas show higher apparent resolution, particularly in the lower bainite specimens, than do the cast Parlodion–silica replicas. This is due to the fact that the silica coating did not develop as much contrast in the fine detail of the structure as did shadowcasting so that this fine detail cannot be observed. This demonstrates that the resolution of a replica method depends on its ability to develop contrast in the fine detail and indicates that the high resolution of silica replicas apparently cannot be utilized in the study of very fine carbides in steels.

Intermediate shadowed Parlodion–silica replicas are equivalent in interpretation and resolution to shadowcast replicas, as the silica coating serves mainly as a support for the shadow film. The silica replica has greater stability in the electron beam.

The subcommittee considers shadowcast plastic replicas somewhat easier to prepare than silica replicas due to fewer steps in the procedure. This is especially true when molded polystyrene is used for the first impression in the silica method.

The molded polystyrene technique has several serious disadvantages. First, there is danger of damaging some specimens by tempering during molding; secondly, fine carbides standing in relief on the surface may be broken off or otherwise distorted by the pressure of the molding operation; and, thirdly, distortion of the polystyrene resulting from undercutting limits the method to extremely light etches. Apart from these practical disadvantages, there remains the serious difficulty of interpreting complex structures.

CONCLUSIONS

After comparing the results of using the two replica methods on the coarse pearlite and lower bainite test specimens, the subcommittee has decided to continue to use the shadowed plastic method for its cooperative program. The intermediate shadowed cast Parlodion–silica replica is considered equivalent, both in interpretation and in resolution, to shadowed plastic replicas and, because of ease in handling, this technique will probably be used by more subcommittee members in the future.

These replicas are easy to interpret and resolve all but the very finest detail. The etch depth used is comparable to that used for high magnification light metallography, and the electron microstructures can be fairly well correlated with the light optical microstructures.

The interpretation of unshadowed silica replicas is very difficult, and the useful resolution of detail in the structures was found to be no better than that of shadowed plastic replicas. Artifacts, such as granularity and distortion resulting from stripping the molded polystyrene from undercut carbides, were difficult to avoid.

It is emphasized that the subcommittee is trying to achieve reproducible electron micrographs of the various steel structures that can be correlated with all the information accumulated over the years with the light microscope. These electron micrographs can also serve as a starting point for research in ferrous metallurgy, using the electron microscope, and as a link between the gross microstructures of steel and very fine detail that may be revealed in the future by the use of new techniques.

APPENDIX TO PART I: DETAILS OF SHADOWED PLASTIC AND PLASTIC–SILICA REPLICA PROCEDURES

Shadowed Plastic Procedure

1. (*a*) The plastic solution is flowed over the prepared surface and dried after the excess has been drained off. Typical plastic solutions used are:

(*1*) Fresh 0.75 per cent solution of formvar in dioxane or ethylene dichloride,

(*2*) 1 per cent Parlodion, or collodion in amyl acetate, or

(*3*) 2 per cent ethyl cellulose in ethylene dichloride.

(*b*) The replica film is removed by moistening its surface with breath and then picking it up with scotch tape upon which a specimen screen has been placed. An alternate method of stripping is to back the replica film with a heavy coating of plastic and then strip the two films together. This procedure avoids strain markings in the replica when stripping from very rough surfaces. Formvar or ethyl cellulose replicas may be backed with Parlodion or Zapon which can be removed by washing in amyl acetate. A 5 per cent solution of polyvinyl alcohol in water can be used to back collodion or Parlodion replicas for stripping and then dissolved away in warm water.

(*c*) The replica is shadowcast by coating the surface with a thin film (10 to 40 Å) of uranium, platinum, or chromium by evaporation from a hot tungsten filament in high vacuum. The metal is deposited at an angle of from $\tan^{-1} 1:2$ to $\tan^{-1} 1:1$ (26½ and 45 deg to the plane of the replica) depending on the size of the detail in the sample. The lower angle is preferable for fine detail, whereas the higher angle is used for coarse structures.

Plastic–Silica Procedure

2. (*a*) *Molded Polystyrene-Silica:*

(*1*) Polystyrene is molded at a pressure of 2000 to 4000 psi and a temperature of 300 F against the prepared surface of the sample. Pressure is applied at 200 F and maintained until the mold has cooled to 150 F. After cooling, the polystyrene is separated from the surface. This step is facilitated by the use of aluminum foil between the sample mounting and the polystyrene with a hole exposing just the specimen.

(*2*) The plastic first replica is coated with a thin film (200 Å) of silica evaporated normally to the surface from a tungsten basket.

(*3*) The silica film is stripped from the polystyrene by immersion in ethyl bromide containing 5 to 10 per cent benzene.

(*4*) The stripped silica films are washed by transferring to a series of wash baths of a 10 per cent solution of isoamyl alcohol in ethyl bromide.

(*5*) The silica replicas made from molded polystyrene negatives are mounted by fishing for them in the final washing bath and capturing them on specimen screens. Rapid evaporation of the solvent, resulting in moisture condensation or rupturing of the replica, is avoided by slow drying in a saturated atmosphere of the bath solvents at a temperature just above the boiling point of the solvent.

(*b*) *Cast Plastic-Silica:*

(*1*) A thick film of Parlodion or Zapon is cast on the prepared surface by solvent evaporation.

(*2*) The replica film may be stripped with scotch tape or just pulled off with tweezers.

(*3*) The replica surface of the film is coated with 200 Å of silica by vacuum evaporation as in the molded polystyrene technique.

(*4*) The Parlodion or Zapon first replica is removed by washing in amyl acetate or ethyl acetate.

(*5*) The silica replicas are mounted by fishing for them and capturing them on specimen screens. Rapid evaporation of the solvent, resulting in moisture condensation or rupturing of the replica, is avoided by drying from the final wash at −30 C.

(*c*) *Variations of Silica Procedure:*

(*1*) If the silica replicas have been handled so that the interface side of the replica is known and can be mounted away from the supporting screen, the replica can be metal shadowcast. This step makes interpretation of surface relief clearer.

(*2*) Another variation is to shadowcast the first plastic replica with metal before coating with silica. The two evaporations can be carried out consecutively without breaking the vacuum by using two filaments. This results in an intermediate shadowed negative replica that has the same appearance and interpretation as the usual one-step plastic replica. Very thin replicas that have extremely good stability under the electron beam can be made in this way.

PART II.—ETCHANTS AND ETCH DEPTHS

The results[1, 2] published by Subcommittee XI as being representative of various microstructures in eutectoid steel have been prepared almost entirely using 4 per cent picral as the etchant. Furthermore, picral was recommended as the preferred etchant for most microstructures in an earlier summary of techniques.[5] This conclusion was reached after investigating the results obtained with several of the more common etchants for steel. Included in the etchant study were:

(*1*) *Picral.*—4 per cent picric acid in ethanol or methanol.

(*2*) *Nital.*—1 to 2 per cent concentrated nitric acid in ethanol or methanol.

(*3*) *Nital–Zephiran Chloride.*—Nital, plus about 0.5 per cent zephiran chloride.

(*4*) *Acid Ferric Chloride.*—2 per cent ferric chloride, 1 per cent hydrochloric acid in ethanol or water

(*5*) *Electroetching Solution.*—10 per cent perchloric acid (70 per cent), 30 per cent water, and 60 per cent ethanol.

Electrolytic etching was examined to a very limited extent. Not only was the effect of the different etches on the development of microstructures examined, but the effect of etch depth was also investigated.

Electron metallography and light metallography present a fundamental difference in the effect of etchants on a metal microstructure. In light metallography, a structure is revealed by differences in the light reflectivity of various constituents. This can be the result of either slightly dissolving certain constituents or of staining them. In electron metallography, structure is observed only through differences in elevation owing to the differential attack of certain constituents by the etchant. Staining plays no part, though it may interfere with the removal of the replica.

Of the etchants studied, picral appears to be the least likely to leave etching residues on the surface. This is desirable since some reagents do tend to leave etching products on the surface. These products are removed as opaque particles by the replicas and interfere with the proper interpretation of microstructure.

Representative electron micrographs are included in this portion of the report to illustrate some of the results obtained by Subcommittee XI with the various etchants studied. All electron micrographs are of the 0.87 per cent carbon steel which has been previously described. The replicas were plastic, shadowed with metal at an angle of approximately 45 deg.

Certain structures present a similar, easily interpreted appearance after treatment with any of several etching agents. Coarse pearlite, for example, has the appearance shown in Fig. 9 when etched with picral. The lamellar carbide is readily seen to project from the surrounding ferrite. Quite similar results are obtained with ferric chloride–hydrochloric acid as the etchant, Fig. 10, although the ferrite in some areas is at-

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[5] "Electron Microstructure of Steel, Appendix," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 489 (1950).

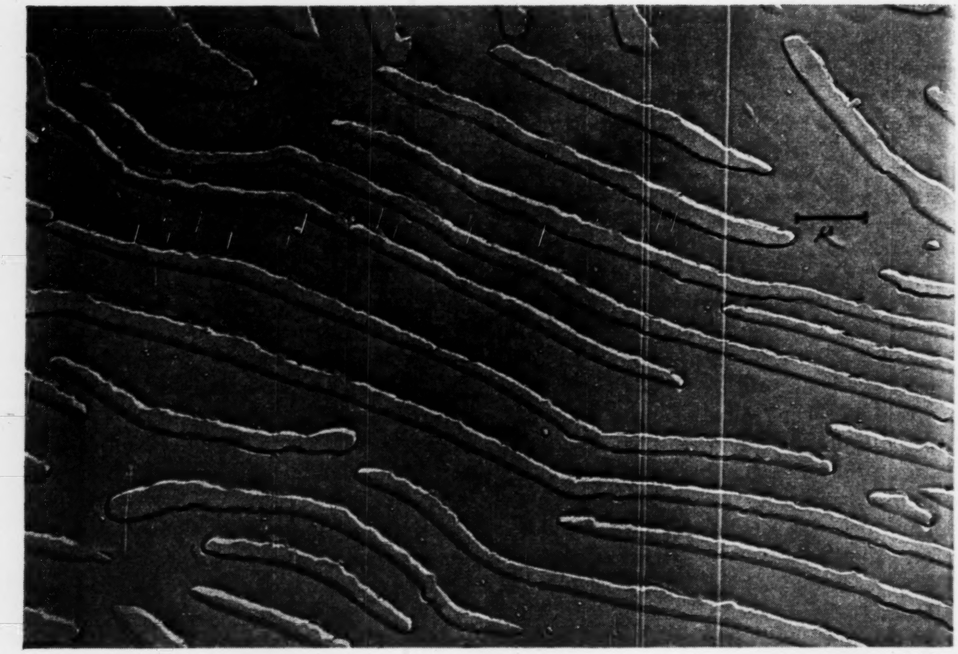


FIG. 9.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F, Picral Etch (× 10,000).

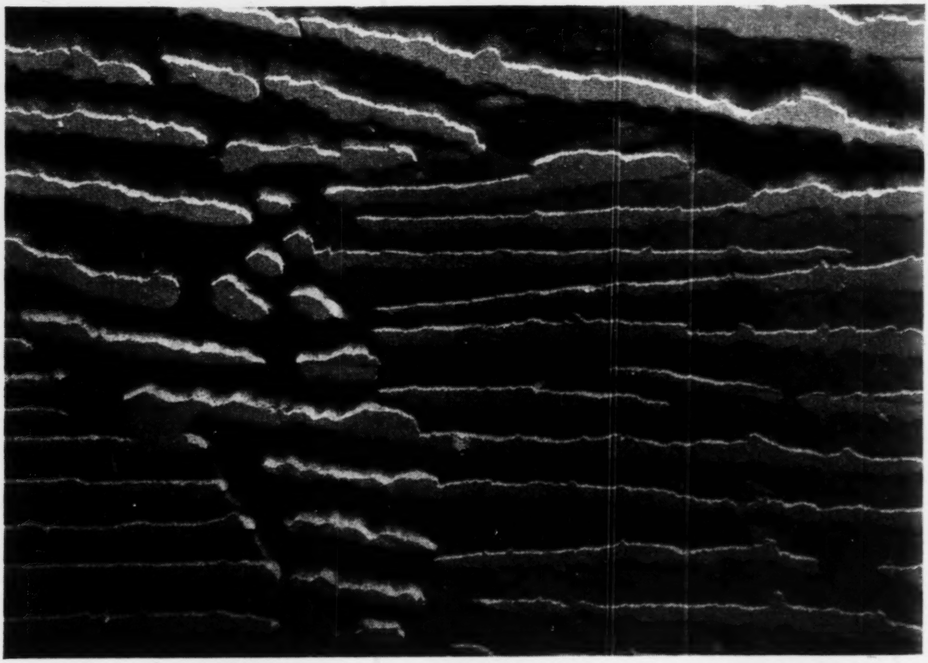


FIG. 10.—Electron Micrograph of Coarse Pearlite, Acid Ferric Chloride Etch (× 10,000).

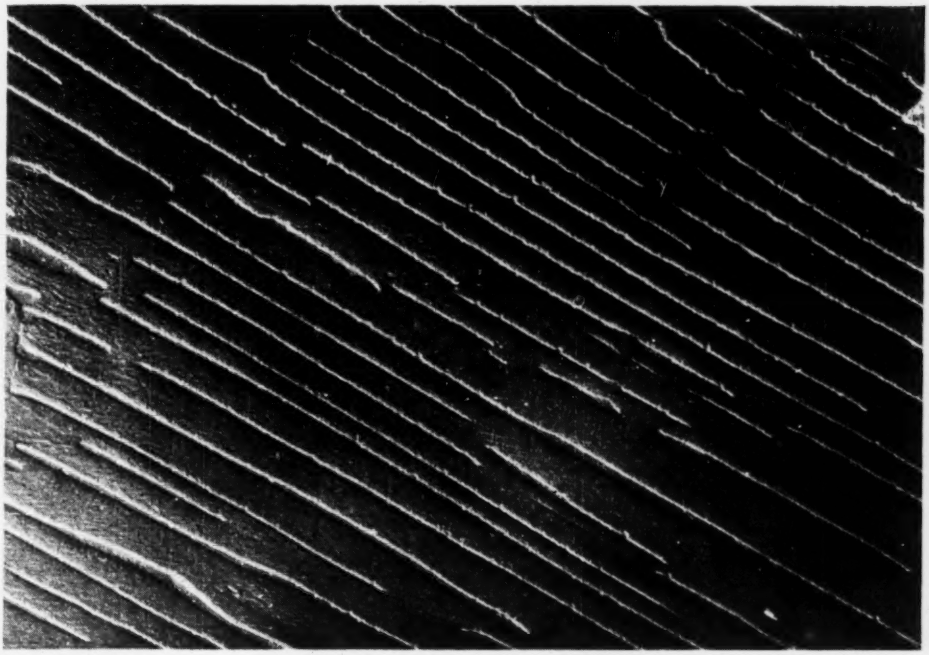


FIG. 11.—Electron Micrograph of Coarse Pearlite, Electrolytic Perchloric Acid Etch (× 10,000).

tacked much more extensively.[1] The extent of attack on the ferrite probably depends on crystal orientation, since ferric chloride–hydrochloric acid is known to etch preferentially along certain crystallographic planes. Coarse pearlite, when electroetched with a perchloric acid solution, presents the same general appearance, Fig. 11, though here also slightly more detail regarding the ferrite orientation may be seen.

Increasing the depth of etch with any of these agents does not appreciably alter the appearance, though the ratio of carbide to ferrite appears to increase if the carbide is sectioned obliquely.

Most coarse carbide-ferrite structures show the same microstructure appearance after etching with any of several solutions. Figure 12 shows spheroidized carbides produced by extensively tempering martensite (12 hr at 1250 F) after etching with picral. The same structure etched with ferric chloride–hydrochloric acid shows a similar distribution of carbides, although the heavier etch in Fig. 13 has attacked the ferrite appreciably, producing a more confused background. Figure 14 is the same structure after electroetching with a perchloric acid solution. While the carbides are left in relief above the surface, considerable detail in the ferrite is also revealed. The grain boundaries are also sharply delineated.

For the study of simple microstructures of the type just illustrated, the choice of etch is less critical. Treatment with picral yields the least complicated results, with little detailed attack on the ferrite to confuse the shape and distribution of carbides.

When studying finer and more complicated structures, results obtained with different etchants are more variable. For instance, bainite isothermally trans-

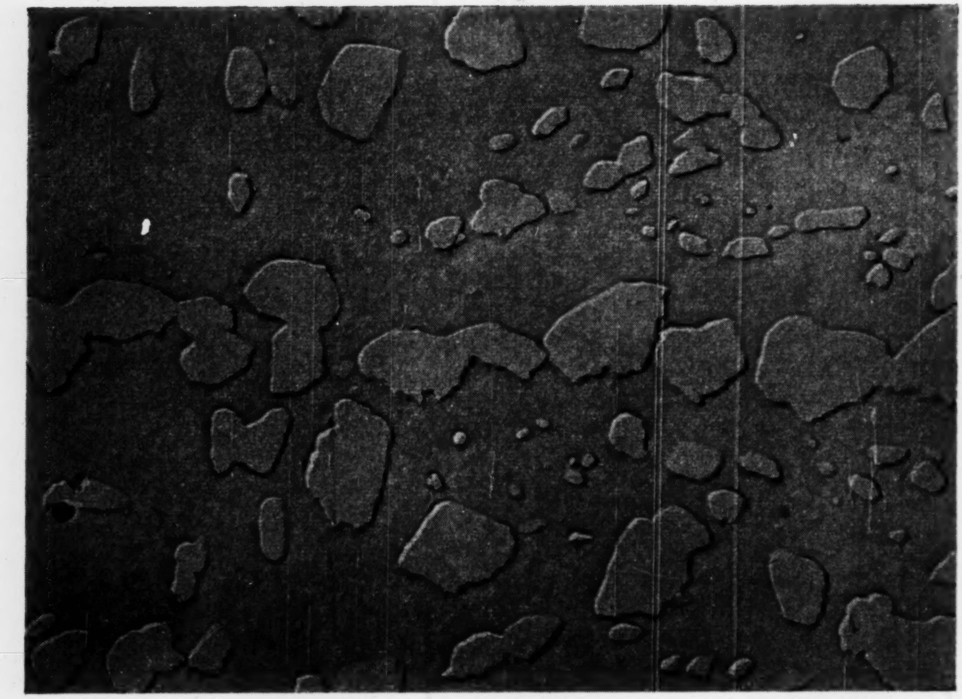


FIG. 12.—Electron Micrograph of Spherodized Carbides, 12 hr at 1250 F, Picral Etch (× 15,000).

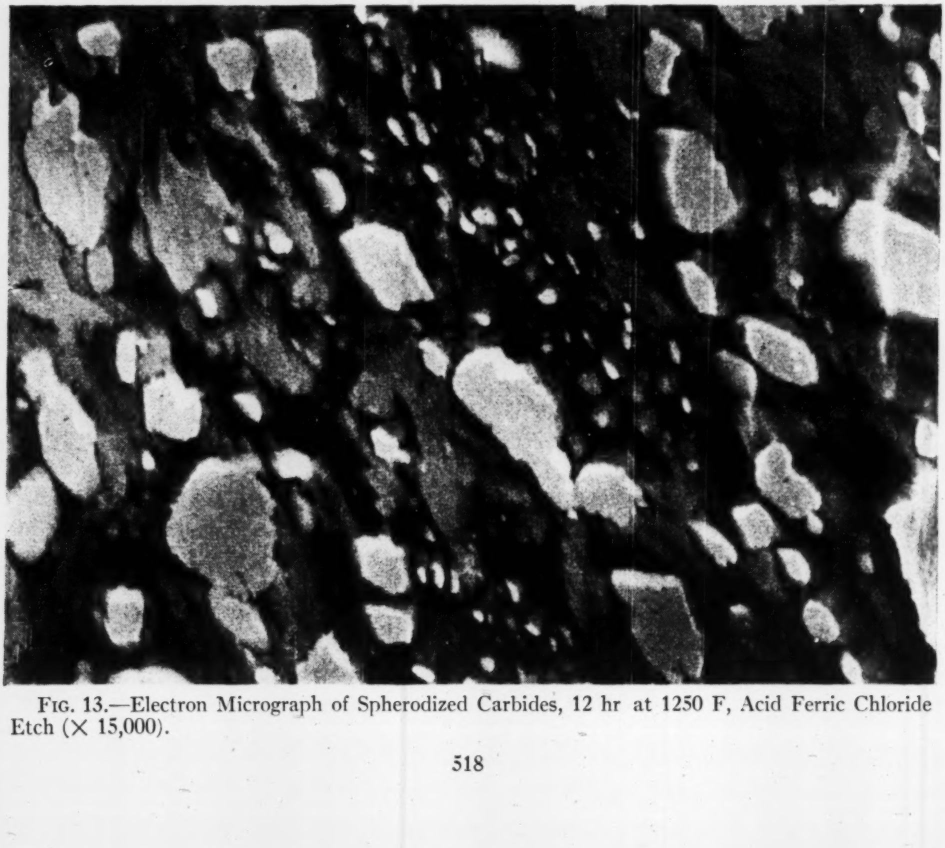


FIG. 13.—Electron Micrograph of Spherodized Carbides, 12 hr at 1250 F, Acid Ferric Chloride Etch (× 15,000).

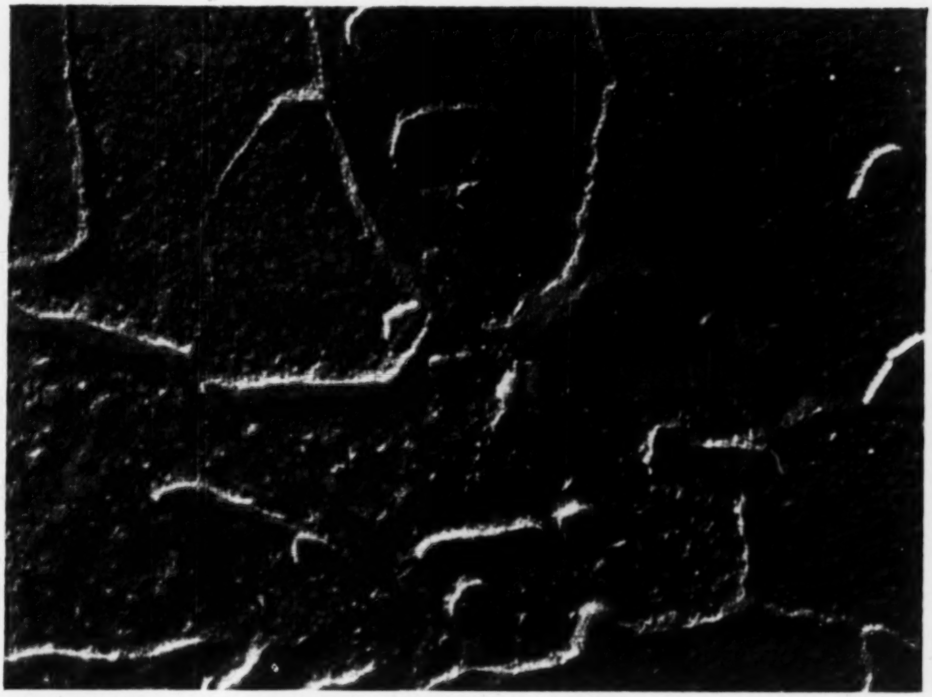


FIG. 14.—Electron Micrograph of Spherodized Carbides, 12 hr at 1250 F, Electrolytic Perchloric Acid Etch (× 15,000).

formed at 550 F contains needles with cross-striated carbide platelets. This characteristic structure is clearly revealed by picral, Fig. 15. Light etching with nital–zephiran chloride yields a similar appearance, Fig. 16, although it is more difficult to control the depth of etch, and some preferential attack of the needles occurs. When this same bainite structure is etched with ferric chloride–hydrochloric acid, the preferential attack on certain ferrite needles is very pronounced, Fig. 17. Although the salient features of the structure remain, the results are more difficult to interpret, especially with the heavy etch illustrated.

Structures of intermediate complexity are also more simply interpreted after etching with picral. Bainite isothermally transformed at 700 F shows the structure illustrated in Fig. 18 after treatment with picral. The shape and orientation of the carbides is readily seen, and the change in carbide shape and distribution is easily followed in a series of specimens transformed at different temperatures.[2] Results nearly comparable to those with picral can be generally attained with dilute nital.

When the same structure (700 F bainite) is etched with ferric chloride–hydrochloric acid, a similar distribution of carbides is revealed (Fig. 19). The distinction between carbide and ferrite, however, is not so clear, and the differential attack on the ferrite in certain areas also complicates the structure. Although some additional information may be provided, the complexity of the microstructure appearance makes more difficult the study of progressive changes with change in transformation temperature.

Extensive etching with nital–zephiran chloride yields the appearance shown in Fig. 20 for the same specimen. The

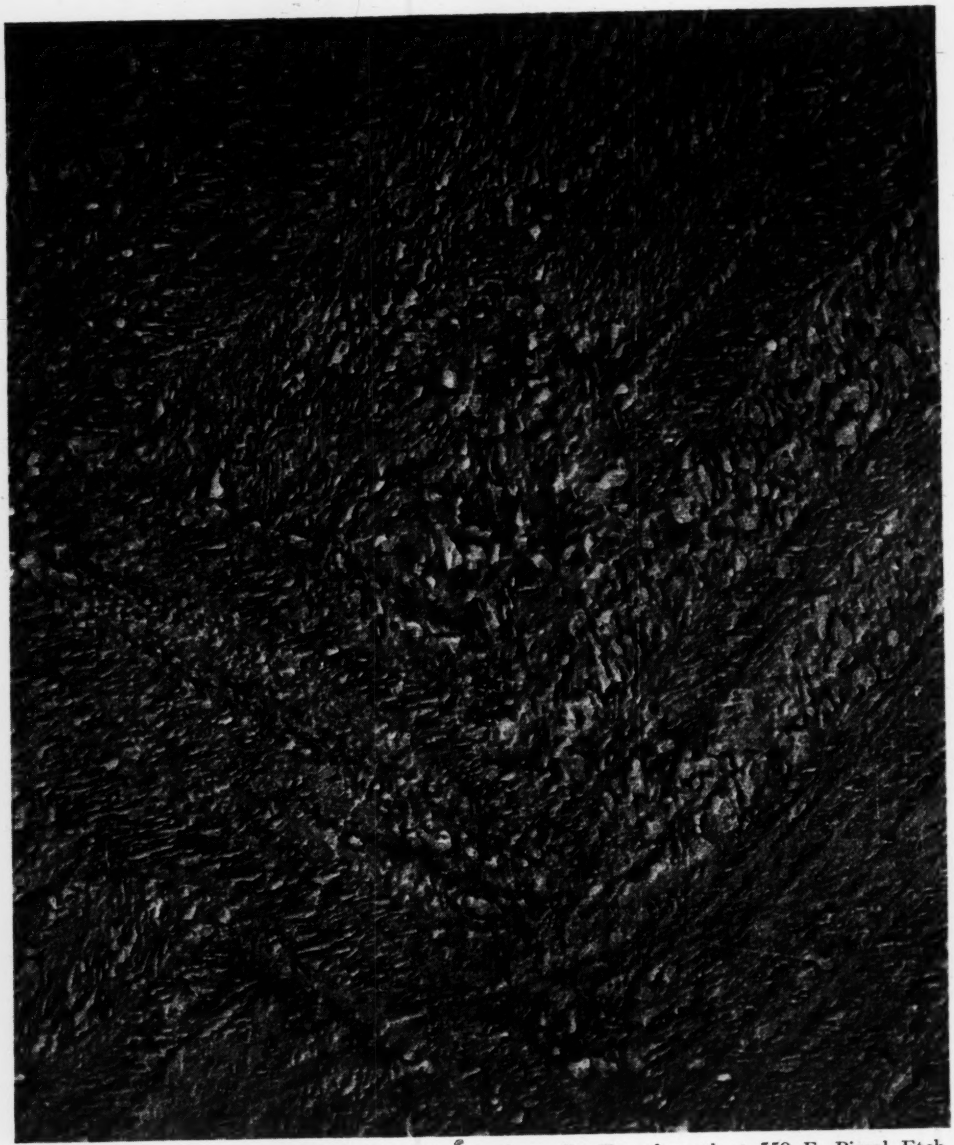


FIG. 15.—Electron Micrograph of Bainite, Isothermally Transformed at 550 F, Picral Etch (× 15,000).

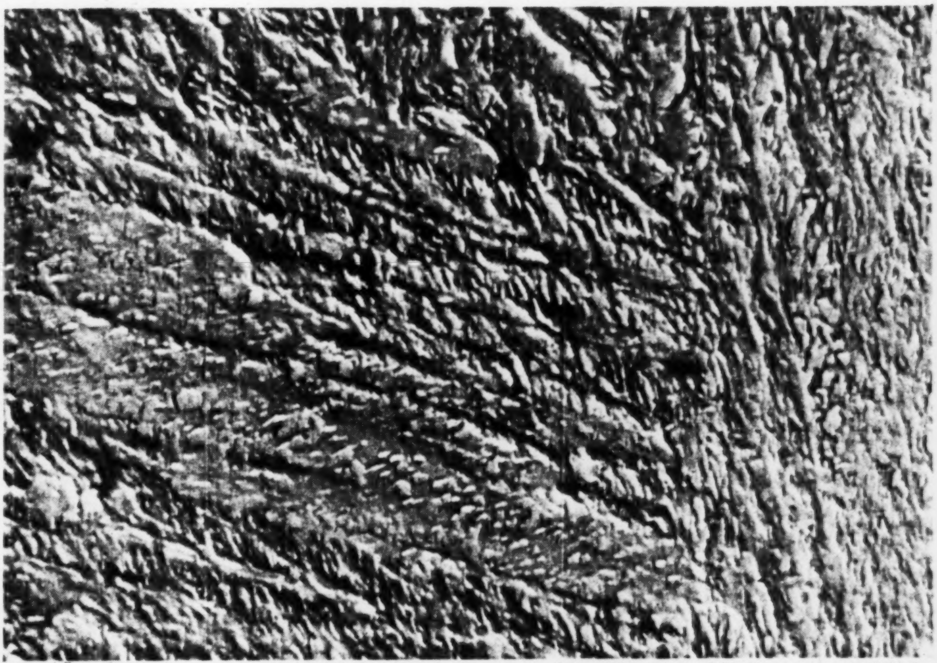


FIG. 16.—Electron Micrograph of Bainite, Isothermally Transformed at 550 F, 4 per cent Nital-1 per cent Zephiran Chloride Etch (8 sec) (× 15,000).

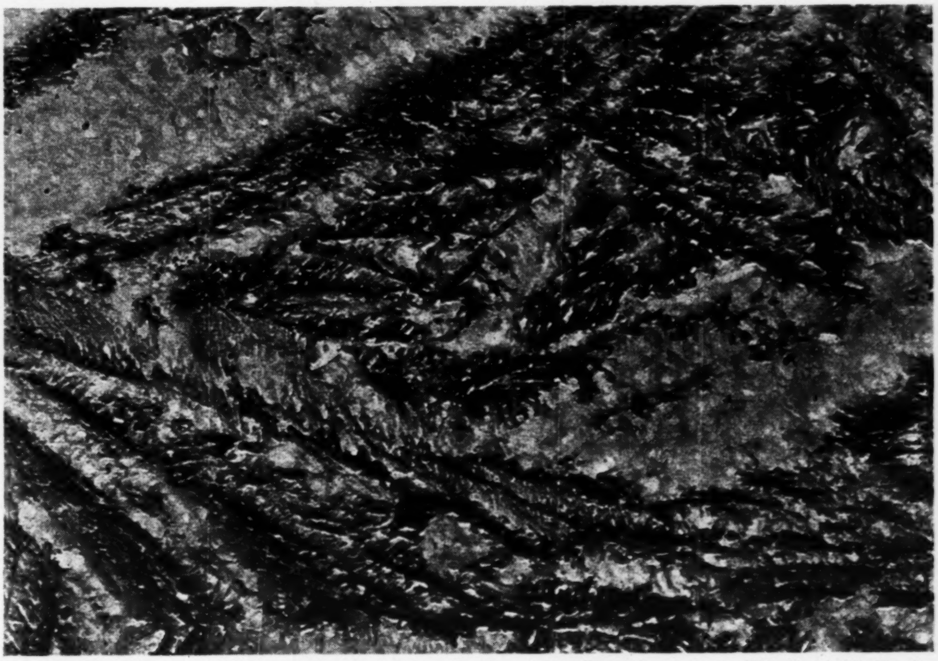


FIG. 17.—Electron Micrograph of Bainite, Isothermally Transformed at 550 F, Acid Ferric Chloride Etch (30 sec) (× 10,000).

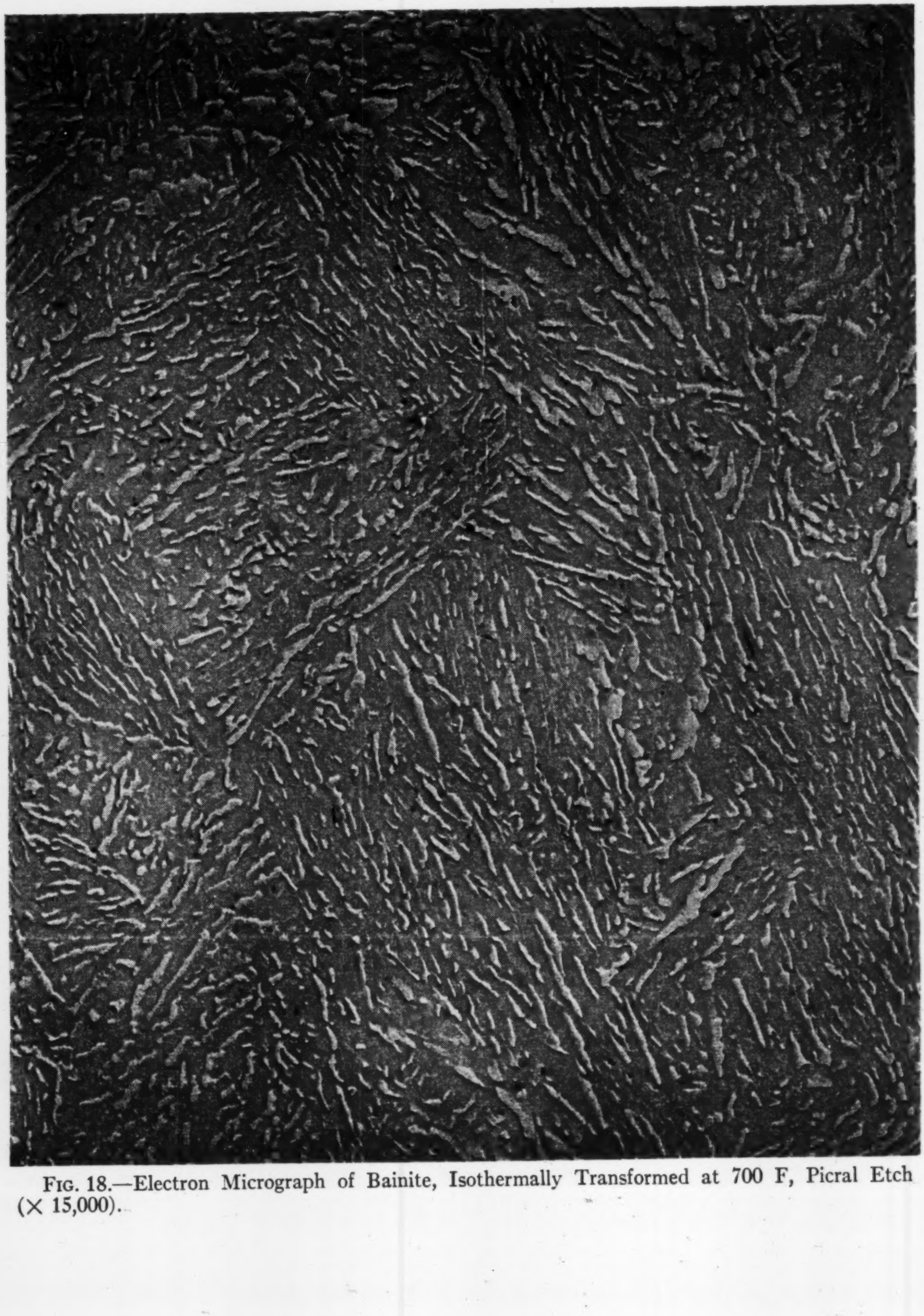


FIG. 18.—Electron Micrograph of Bainite, Isothermally Transformed at 700 F, Picral Etch (× 15,000).

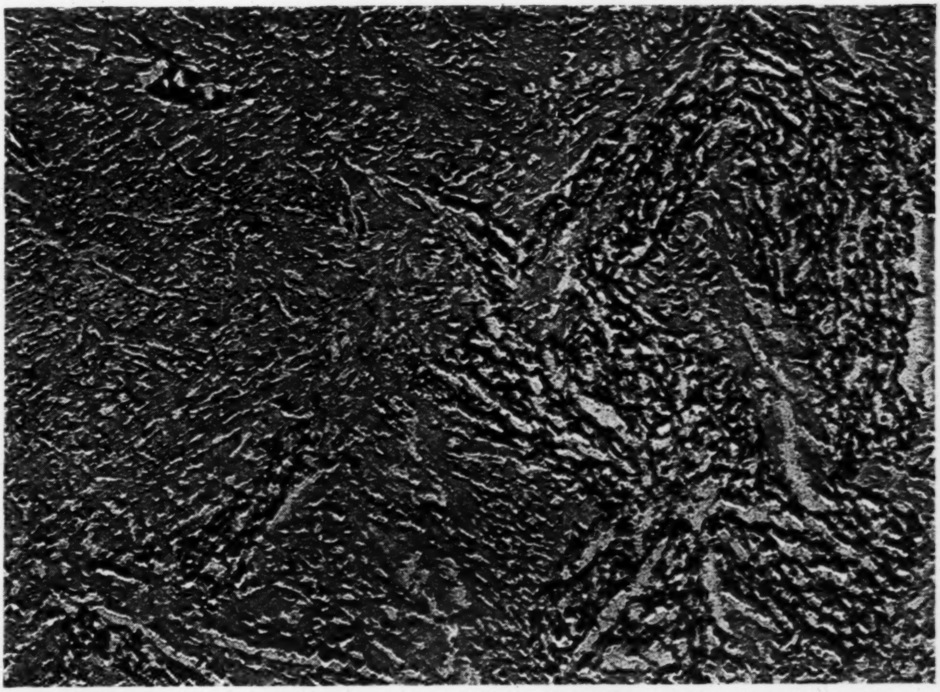


FIG. 19.—Electron Micrograph of Bainite, Isothermally Transformed at 700 F, Acid Ferric Chloride Etch (20 sec) (× 10,000).

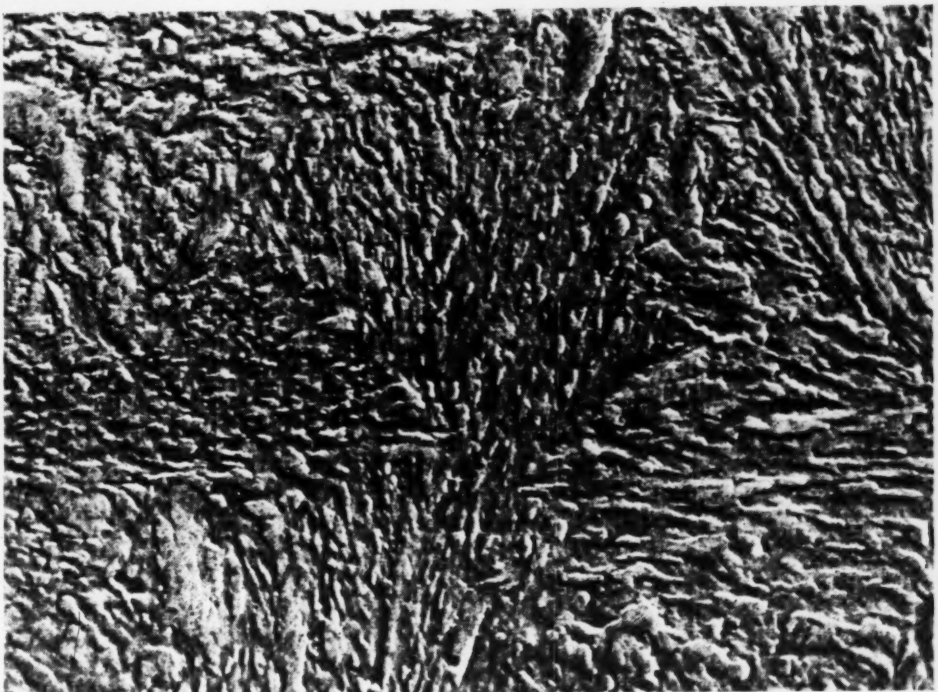


FIG. 20.—Electron Micrograph of Bainite, Isothermally Transformed at 700 F, 4 per cent Nital-1 per cent Zephiran Chloride Etch (40 sec) (× 15,000).

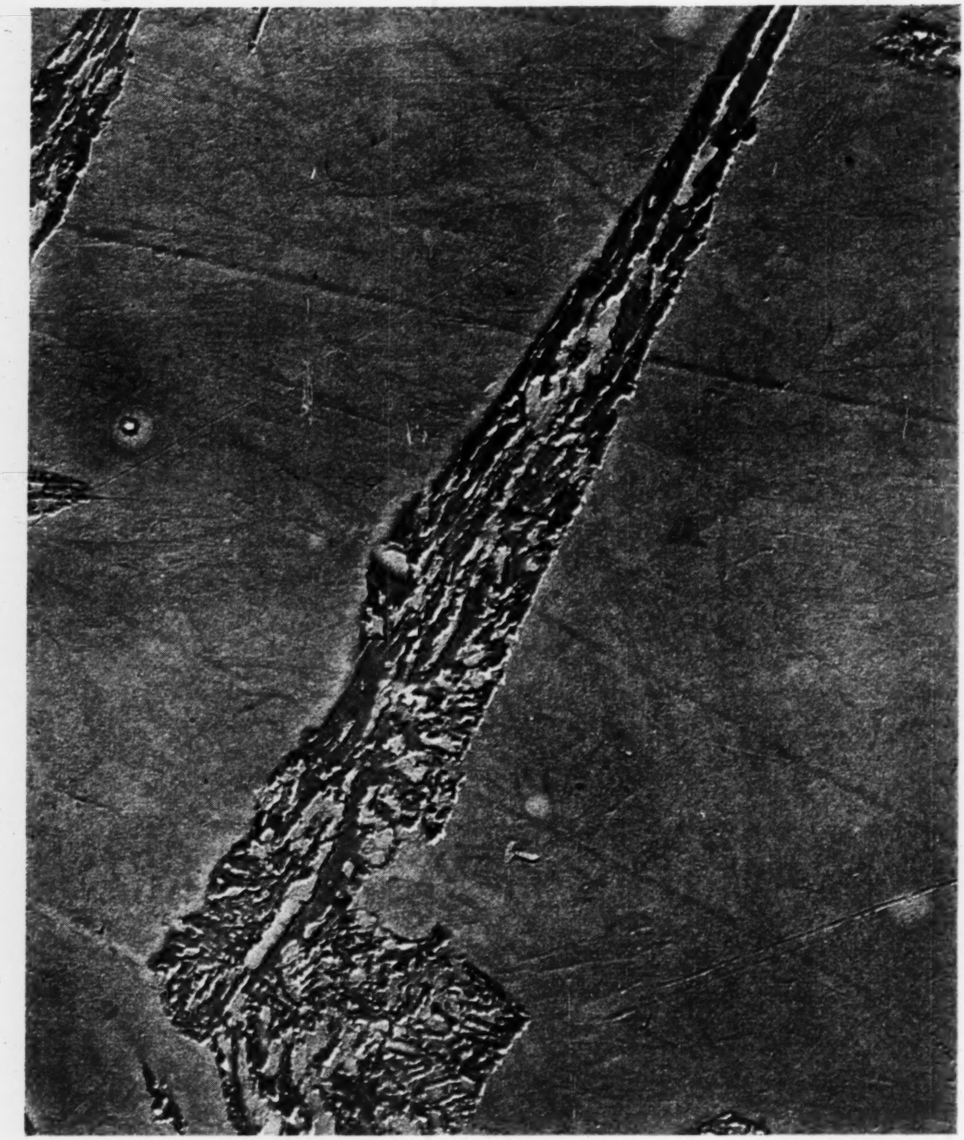


FIG. 21.—Electron Micrograph of Specimen Transformed About 20 per cent to Bainite by Isothermal Treatment, 12 sec at 850 F, Picral Etch (× 10,000).

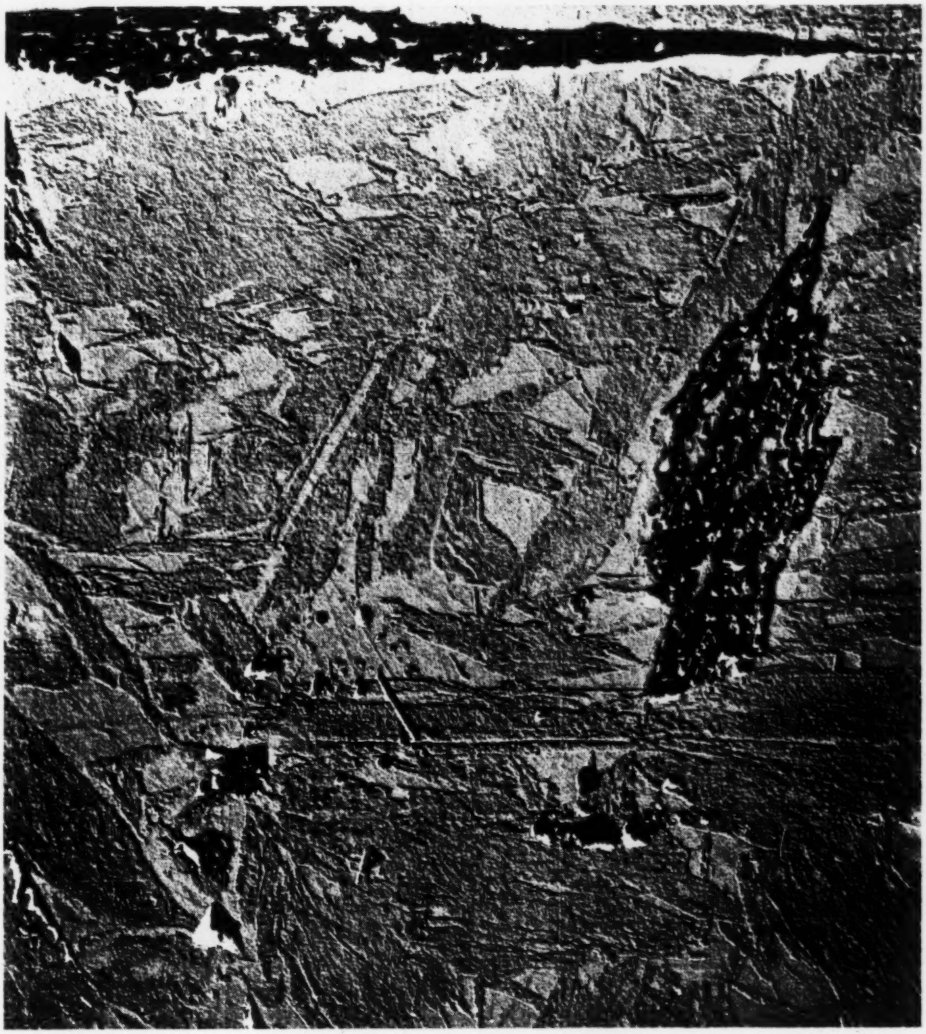
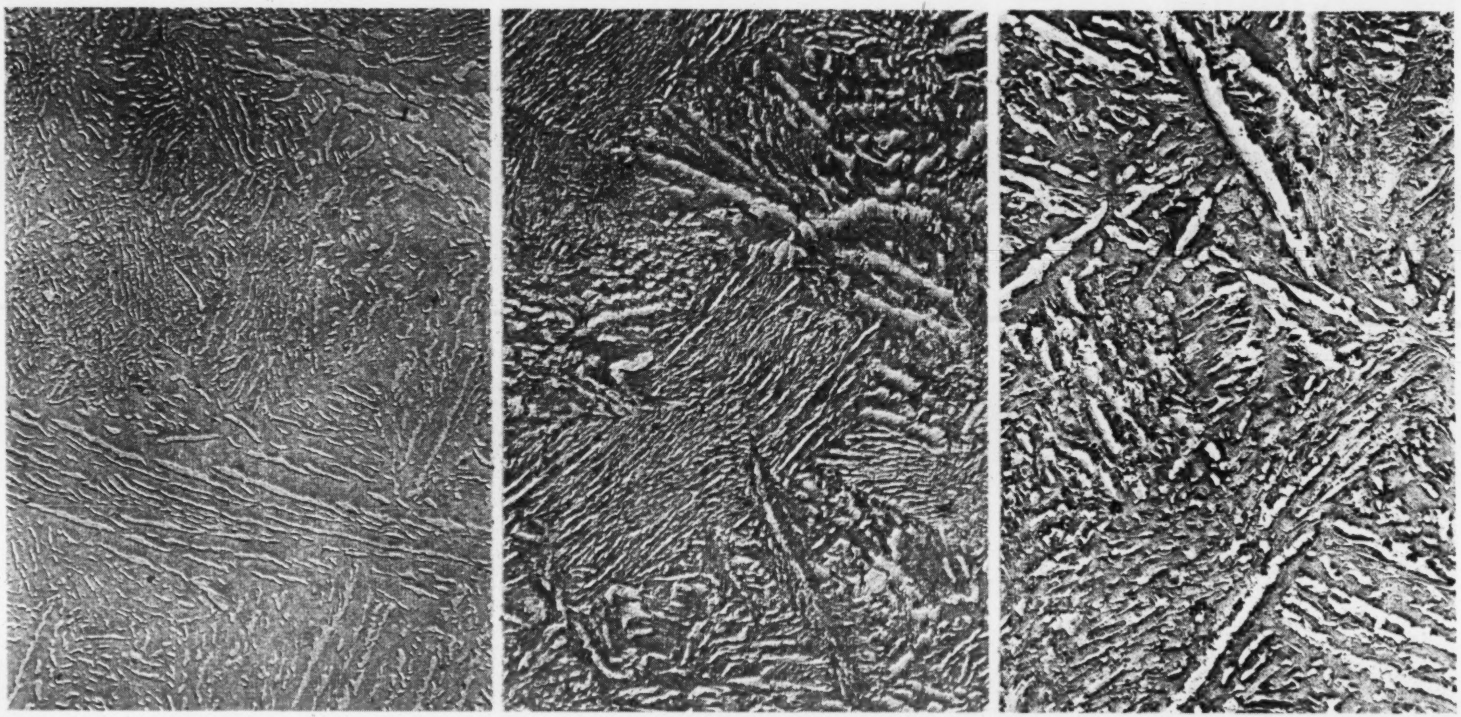


FIG. 22.—Electron Micrograph of Specimen Transformed About 20 per cent to Bainite by Isothermal Treatment, 12 sec at 850 F, Nital-Zephiran Chloride Etch (× 10,000).

structure is overcomplicated and less readily interpreted.

In some instances appreciably more information can be obtained regarding a given structure by using more than one etchant. As an example, an electron micrograph of a specimen transformed 20 per cent to bainite at 850 F is shown in Fig. 21 after etching with picral. Although the details of the carbide and ferrite in the bainite needles are excellently shown, the surrounding martensite structure is unattacked, and no information is available regarding this phase. When the same specimen is heavily etched with nital–zephiran chloride (Fig. 22), the martensite is appreciably etched. Angular areas of retained austenite are revealed as whiter areas that are not attacked by the



(*a*) Picral Etch, 12 sec. (*b*) Picral Etch, 15 sec. (*c*) Picral Etch, 17 sec.

FIG. 23.—Electron Micrograph of Bainite, Isothermally Transformed at 850 F, Effect of Etch Time on Appearance of Carbide Phase (× 7500).

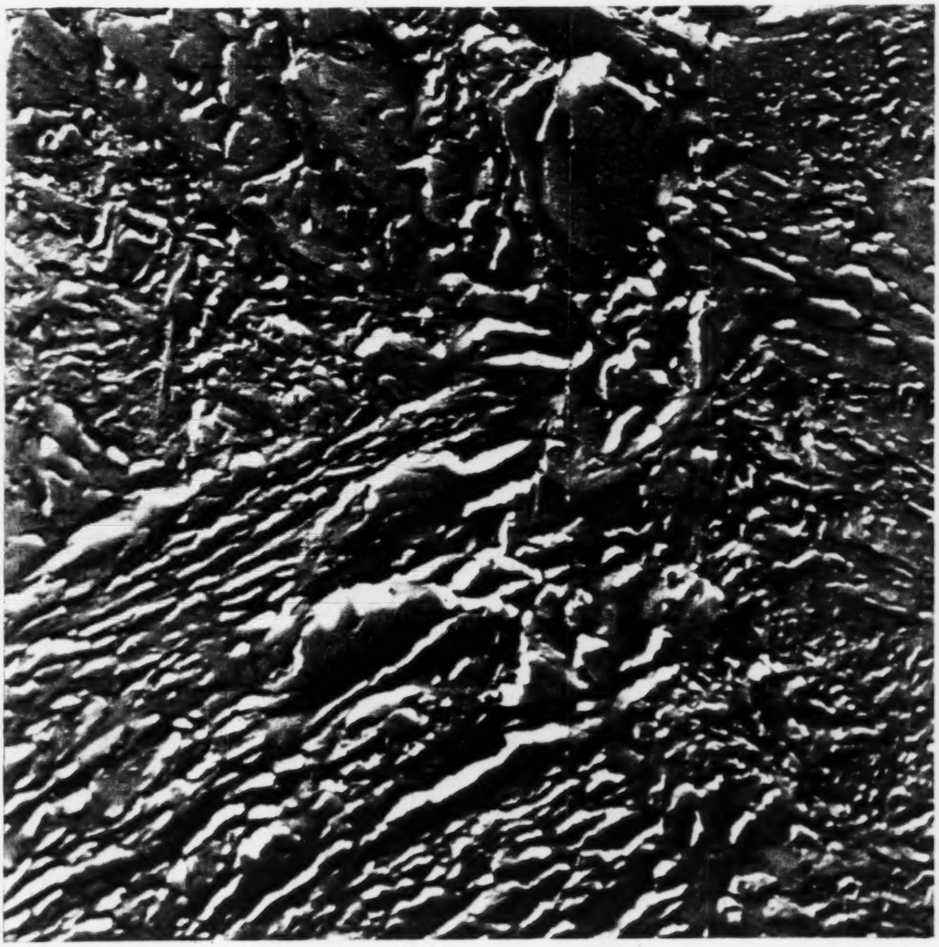


FIG. 24.—Electron Micrograph of Bainite, Isothermally Transformed at 850 F, Showing Platelike Structure of Carbides, Nital-Zephiran Chloride Etch (× 15,000).

etchant. The bainite needles are so deeply etched by this treatment that their structure is obliterated. A good correlation was obtained between the amount of retained austenite visually observed by this technique and X-ray spectrometer results.

The results presented here suggest that more than one etchant may be used to study a given microstructure. Additional research may develop new etchants which will be especially well suited for electron microscopy. Furthermore, some of the details revealed by the etchants that have been discussed may eventually be found to be significant, as the microstructures are more completely understood. Picral has been preferred only because it provides the simplest and clearest delineation of carbide and ferrite phases.

EFFECT OF ETCH DEPTH

In addition to the study of various etchants, the effect of etching time or extent of attack was examined. Initially, a fairly heavy etch was preferred. This was necessary to produce replicas with

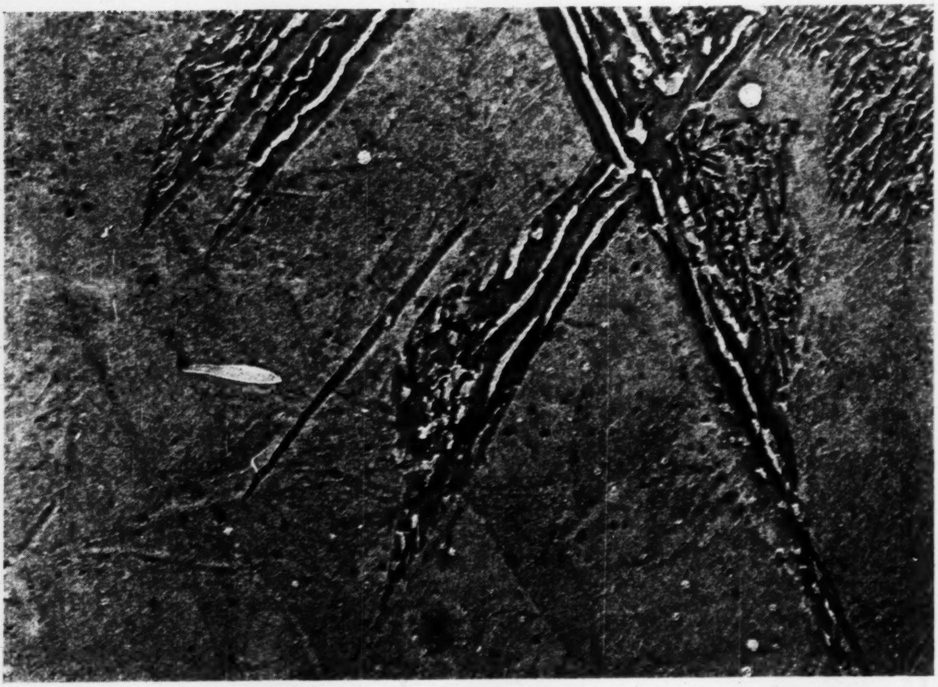


Fig. 25.—Electron Micrograph of Specimen Transformed About 50 per cent to Bainite by Isothermal Treatment, 20 sec at 850 F, Picral Etch (× 15,000).

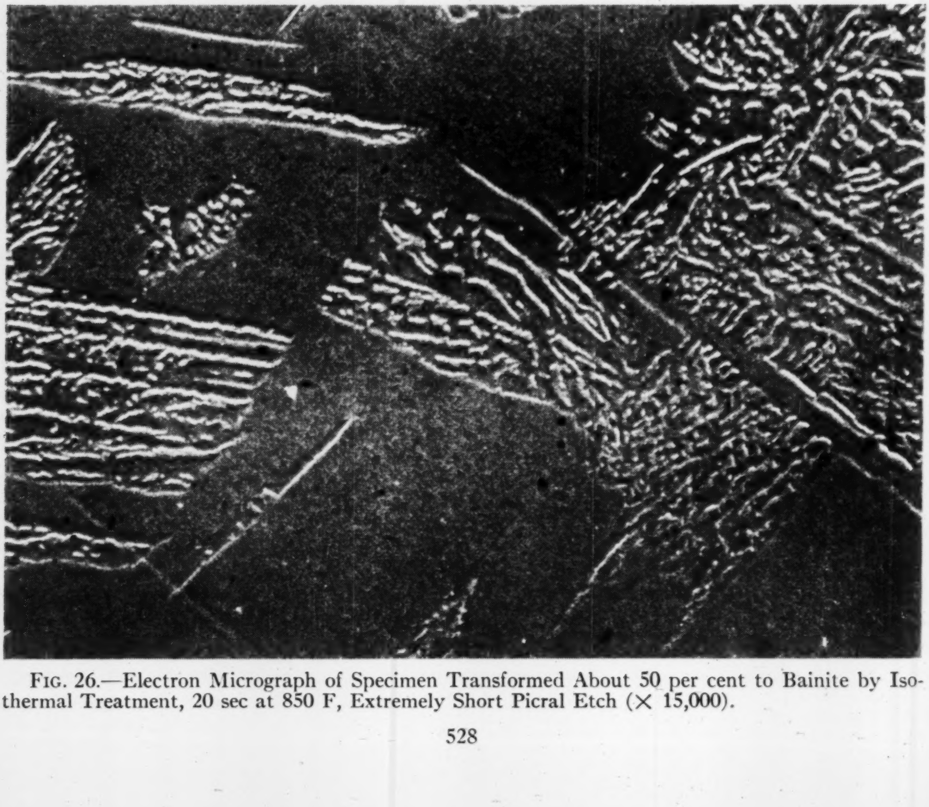


Fig. 26.—Electron Micrograph of Specimen Transformed About 50 per cent to Bainite by Isothermal Treatment, 20 sec at 850 F, Extremely Short Picral Etch (× 15,000).

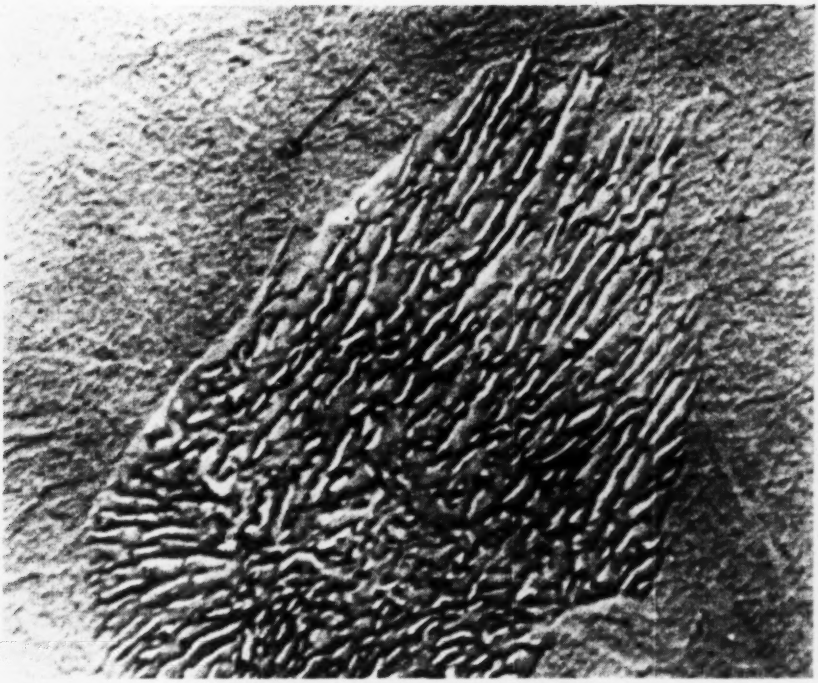


FIG. 27.—Electron Micrograph of Specimen Transformed About 20 per cent to Bainite at 850 F, Moderate Picral Etch (× 15,000).

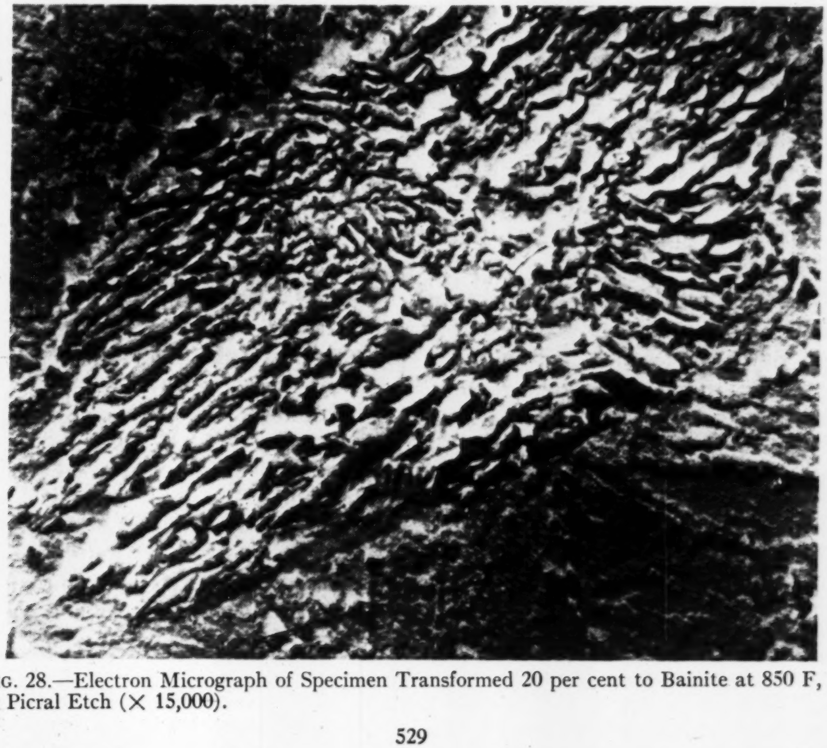


FIG. 28.—Electron Micrograph of Specimen Transformed 20 per cent to Bainite at 850 F, Very Long Picral Etch (× 15,000).

sufficient contrast to define the structure. With the development of metal shadowing and improved replica techniques, however, it has been found that very light etching is usually desirable. Light etches do not obscure fine structures, and they give a more correct representation of the size and relative amount of carbide. Occasionally, heavier etches are employed to reveal certain features of a microstructure.

One of the hazards of overetching is that illustrated in a series of electron micrographs, Fig. 23. These micrographs are all of bainite isothermally transformed at 850 F. Figure 23(*a*) is a micrograph of the specimen etched lightly with picral. A progressive increase in etching, Figs. 23(*b*) and (*c*), exposes more of the carbides, making them appear larger. This precludes studying the progressive changes in carbide size and shape in specimens transformed at different temperatures unless a minimal etch depth is used. The simple appearance of Fig. 23(*a*) is also more readily understood than Fig. 23(*c*).

On the other hand deliberate overetching may sometimes reveal additional detail. Figure 24 is a micrograph of the same specimen just discussed, but here extreme etching indicates that some of the carbides formed at this temperature are plate-like, similar to pearlite.

The differences which may be encountered by etching to various degrees with the same etchant are also illustrated in the electron micrographs, Figs. 25 to 28. Figure 25 shows the structure of bainite transformed 50 per cent at 850 F, after suitable etching with picral. The orientation of carbide and ferrite within the needles is readily observed, including some needles which are sectioned so that only ferrite is seen. Figure 26 shows the results of too light an etch. The structure is obscure because the carbide and ferrite are not adequately distinguished.

As an example of the complications produced by overetching, electron micrographs show a bainite needle slightly overetched (Fig. 27) and exactly the same area after a prolonged etch (Fig. 28). While the typical carbide-in-ferrite structure of the bainite needle may be seen in Fig. 27, only a disorganized mass of carbides remains of the same bainite needle in Fig. 28.

These illustrations indicate that a light etch is generally desirable for the examination of microstructures with the electron microscope.

CONCLUSIONS

The examination of various microstructures in eutectoid carbon steel with the electron microscope, using different etchants and etch depths, suggests the following general principles:

(*1*) Etching with 4 per cent picral produces the simplest and clearest delineation of carbide and ferrite phases. It leaves a minimum of staining and etch products on the surface.

(*2*) Acid ferric chloride and, to a lesser extent, nital–zephiran chloride, preferentially attack the ferrite in some areas. This produces a more complicated microstructure, though it may provide some information regarding ferrite orientation.

(*3*) Limited experience suggests that electrolytic etching produces a clean surface, with some indication of ferrite orientation and grain boundaries.

(*4*) A heavy nital–zephiran chloride etch is useful to reveal retained austenite in martensite.

(*5*) A light etch is preferable for general microstructure study since it is more easily interpreted and provides a more accurate representation of carbide and ferrite phases.

(*6*) Heavier etches are sometimes useful for showing structure features, such as the shape of carbides.

REPORT OF COMMITTEE E-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTIONS*

Committee E-5 held one meeting during the past year, in Detroit, Mich., on March 5, 1953. Meetings of subcommittees preceded this meeting.

The Committee consists of 47 members, of whom 21 are classified as producers, 6 as consumers, and 20 as general interest members, with 5 consulting members.

A revised scope statement stating more clearly the over-all activities of the committee was approved by the board of directors of the Society.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends that the following revision of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 50**)[1] be approved for reference to letter ballot of the Society for immediate adoption and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Section 10.—Delete the last phrase of the first sentence reading: "this will usually require about 30 days time under favorable drying conditions," and add the following:

> Wherever practical, the material or construction shall be weighed at intervals during the period of drying and the weights on successive days recorded. Weighing shall be continued until the weight shall have remained fundamentally constant for not less than 5 successive days under favorable drying conditions of the open room of the Laboratories. If weighing of the total material or construction of the test specimen is impractical, a suitable sample designed to give an accurate estimate of the condition of dryness of the complete sample shall be selected or fabricated and subjected to weighing as specified above for the complete test specimen.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 50**), issued June, 1950, covering the incorporation of new Sections 27 to 30[2] covering an Alternate Test of Protection for Structural Steel Beams and Girders be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVE REVISION OF STANDARD

The committee recommends the following tentative revision of the Standard Methods of Fire Tests of Building Construction of Materials (**E 119 – 50**):[1]

Section 6.—Delete the two Paragraphs (*a*) and (*b*) which read as follows:

> (*a*) The test structure may be located at any place where all the necessary facilities for properly conducting the test are provided.
>
> (*b*) Entire freedom is left to each investigator in the design of the test structure and the nature and use of fuel, provided the test requirements are met.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

[1] 1952 Book of ASTM Standards, Part 4.

[2] 1952 Book of ASTM Standards, Part 4, p. 1003.

Section 9.—Add the following line to Table I referred to in Paragraph (*d*) under the column headings Resistance Period, Water Pressure, Duration, respectively: "Less than one hour if desired," "30 psi," "1 min."

Section 26.—Delete Paragraph (*b*), which reads as follows:

> (*b*) The construction shall have sustained the applied load during the fire and hose stream test as specified in Section 9, without passage of flame, of gases hot enough to ignite cotton waste, or of the hose stream. After cooling, but within 72 hr. after completion of the hose stream test, floor specimens shall sustain the dead load of the test construction plus twice the superimposed load specified above.

WITHDRAWAL OF TENTATIVE REVISION

The committee recommends the withdrawal of the following tentative revision of the Standard Methods (**E 119 – 50**), issued June, 1950:

Section 26(c).—Change lines 5 and 6 to read "More than 250 F (139 C) in case of floors and 400 F (222 C) in case of roofs, above the initial temperature."

TENTATIVE AND STANDARD CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Fire Hazard Classification of Building Materials (**E 84 – 50 T**)[1] be continued as tentative without change, pending possible revision resulting from research now being conducted upon smaller tunnel-type test equipment.

The committee recommends that the Standard Methods of Fire Tests of Door Assemblies (**E 152 – 41**)[1] be continued as standard without change, pending further study.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

A special committee prepared a draft of a fire test method using small size specimens as suggested by ASTM Committee E-6 on Methods of Testing Building Constructions for inclusion as an appendix to Standard Methods E 119. This report was circulated for letter ballot to the entire committee. Several negative ballots were received with suggested changes and comments. These comments will be reviewed by the special committee, appropriate changes made, and a revised draft submitted to Subcommittee I.

Subcommittee I on Fire Tests of Materials and Construction (G. N. Thompson, chairman).—Section 10 of Methods E 119 – 50, having to do with the conditioning of specimens, is to receive further study, particularly as to the requirements for artificial drying of the specimens. The requirements of Methods E 119 – 50 that test assemblies shall be termed "combustible" if a test assembly ignites or continues to flame after the furnace fire is shut off, is being given further consideration in order more fully to define the use of the term "combustible," together with the fire resistance rating assigned to the specimen.

Subcommittee III on Fire Tests of Wall Opening Assemblies (H. D. Foster, chairman) has been active on the review of the Standard Method of Fire Tests of Door Assemblies (E 152 – 41). A second draft of a complete revision of the method, incorporating advisable changes, is being prepared.

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes.—The investigational work using a smaller size tunnel test equipment than that specified in Tentative Method of Fire Hazard Classification of Building Materials (E 84 – 50 T) has been retarded due to changes in committee membership and lack of funds. Industry is to be solicited for funds and further tests conducted to obtain greater perfection in the control of draft conditions and observations of results using the small tunnel test equipment.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Nomenclature and Definitions (J. R. Shank, chairman).—Good progress is being made in the preparation of a proposed tentative method of tests for defining the term "noncombustible" as applied to building material. The test method will provide technical and scientific knowledge of the combustibility of homogeneous materials having very low ranges of combustibility and of such nature that they could be tested by this method.

Subcommittee VIII on Fire Tests of Roof Coverings (A. J. Steiner, chairman).—Several drafts of a proposed test method for establishing the fire hazard of roof coverings have been prepared and subsequently revised as dictated by committee discussions. Certain features pertaining to materials and their method of application are under discussion with representatives of the roofing industry. The results of these discussions will expedite the preparation of a final draft of the test method.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 37 returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. L. Brown,
Chairman.

R. C. Corson,
Secretary.

REPORT OF COMMITTEE E-7
ON
NON-DESTRUCTIVE TESTING*

Committee E-7 on Non-Destructive Testing met at New York, N. Y., on June 25, 1952, in conjunction with the Annual Meeting of the Society. Numerous Executive Council and subcommittee meetings have been held since.

The committee is planning a technical session to be held Thursday, July 2, 1953, during the Annual Meeting, in addition to the usual committee meetings. It is planned that a number of papers on correlation of non-destructive tests with each other and particularly with product performance will be presented.[1]

The membership of the committee is now 104, plus 18 consulting members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee E-7 presented to the Society through the Administrative Committee on Standards a Tentative Recommended Practice for Radiographic Testing. The recommended practice was accepted by the Standards Committee on September 5, 1952, and appears in the 1952 Book of of ASTM Standards, Parts 1 and 2, bearing the designation E 94 – 52 T.

NEW TENTATIVE

The Bureau of Aeronautics has offered its set of Reference Radiographs for Inspection of Aluminum and Magnesium Castings[1a] to the Society for use as an ASTM standard, if desired. The committee recommends this offer be accepted and the set published as ASTM Tentative Reference Radiographs for Inspection of Aluminum and Magnesium Castings.[2]

The recommendation above has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (**E 52 – 49 T**)[4] be continued for one more year without revision.

ACTIVITIES OF SUBCOMMITTEES

Editorial Subcommittee (S. A. Wenk, chairman).—This new subcommittee was organized during the year and assumed jurisdiction over (*1*) the annual review of all E-7 standards publications, (*2*) the coordination of E-7 activities of primarily editorial nature (preparation of glossaries, etc.), and (*3*) the maintenance of uniform format of E-7 publications.

Subcommittee I on Radiographic Recommended Practice (H. E. Seeman, chairman).—Publication of the Tentative Recommended Practice for Radiographic

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* Presented at the Fifth-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] See ASTM BULLETIN, No. 197, April, 1954, p. 52 (TP80).

[1a] Aeronautical Technical Inspection Manual, Vol. 3, Section 5, Bureau of Aeronautics, Navy Department.

[2] These Tentative Reference Radiographs have been approved by and accepted by the Society in accordance with established procedures, for use pending adoption as standard. A description of these radiographs appears as ASTM Designation E 98 – 53 T, 1953 Supplement to Book of ASTM Standards, Part 2.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] 1952 Book of ASTM Standards, Parts 1 and 2.

Testing (E 94 - 52 T) was completed during the year.

Subcommittee II on Reference Radiographs (Alexander Gobus, chairman).—Final radiographs of specimens for the initial set of Reference Radiographs of Steel Welds are being prepared by Task Group A. Other task groups are working on additional radiographs of submerged arc welds for later incorporation in this set, and on a study of possible need of reference radiographs of thick castings (5 to 20 in. thick). It is planned that papers on all three projects will be offered at the E-7 technical session at the time of the Annual Meeting.

Subcommittee III on Magnetic Particle and Penetrant Testing (H. Migel, chairman).—A final draft of the proposed Tentative Method for Dry Powder Magnetic Particle Testing is being prepared for presentation to the main committee.

Subcommittee IV on Technical, Economic, and Application Data (R. C. McMaster, chairman).—The scope and functions of this subcommittee are being re-evaluated by the Executive Council.

Subcommittee V on Radiographic Procedure (C. H. Hastings, chairman).—An investigation of penetrameter types is being carried out in the hope of developing a type which might be suitable for indicating over-all radiographic quality.

Subcommittee VI on Ultrasonic Testing (J. C. Smack, chairman).—Final drafts of two proposed Tentative Recommended Practices for Reflection and Resonance Testing, and of proposed Tentative Specifications for Reference Blocks for Ultrasonic Testing are being prepared for submission to the main committee.

This report has been submitted to letter ballot in the committee, which consists of 104 members; 66 members have returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

D. T. O'CONNOR,
Secretary

REPORT OF COMMITTEE E-9

ON

FATIGUE*

During 1952, *STP No. 121* "Symposium on Statistical Aspects of Fatigue," covering papers presented in 1951, was published. This has been followed by *STP No. 137* "Statistical Aspects of Fatigue—II," covering the papers presented in 1952. A Task Group under J. T. Ransom is attempting to formulate statistical procedures which will be useful in planning and interpreting fatigue investigations.

Subcommittee I (*Research*) (H. F. Moore, chairman) has discussed current research and summarized the problems of main interest in minutes. Those interested may obtain a copy of the summary by writing to the chairman.

Subcommittee II (*Papers*) (W. T. Lankford, chairman) has screened a considerable number of papers during the year. Two sessions on fatigue have been developed for the 1953 Annual Meeting.

Subcommittee III (*Survey*) (T. J. Dolan, chairman) has provided another collection of "References on Fatigue," this issue (*STP No. 9-C*) covering articles published in 1951. Multilith copies may be purchased through Headquarters at a nominal price.

Subcommittee IV (*Large Machines*) (J. M. Lessells, chairman) has been active with regard to the marine propeller shaft problem. Some tests will probably be undertaken by the Timken laboratory under sponsorship of the American Bureau of Shipping and the American Society of Naval Architects.

A brief report of the fatigue projects of the Mechanical Engineering Research Laboratories at East Kilbride, Scotland, was presented before the committee by C. E. Phillips of MERL.

Committee E-9 participated in a Panel Discussion on "Fatigue Techniques and Evaluation" sponsored by the Society for Experimental Stress Analysis at Milwaukee, May 21, 1953.

At the biennial election at the June 25, 1952 meeting, the following officers of E-9 were elected: Chairman: R. E. Peterson; Secretary: O. J. Horger; Advisory Committee: J. A. Bennett, T. J. Dolan, G. R. Gohn, L. R. Jackson, W. T. Lankford, J. M. Lessells, H. F. Moore, and R. L. Templin.

During the year the following new members were added to Committee E-9: J. H. Flanagan, Watertown Arsenal (replacing H. C. Mann); C. B. Griffin, General Motors Corp.; Harold Hessing, Bureau of Aeronautics, Navy (replacing E. J. Greene); S. E. Maddigan, Kaiser Aluminum and Chemical Corp.; P. R. Toolin, Westinghouse Electric Corp.; E. H. Schuette, Dow Chemical Co. (replacing G. H. Found); F. B. Stulen, Curtiss Wright Corp. New member added to Subcommittee I Research: E. Orowan, Massachusetts Institute of Technology.

This report has been submitted to letter ballot of the committee, which consists of 48 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectively submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 29–July 3, 1953.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

Committee E-11 was organized in 1946 to promote the knowledge of quality control methods and their application to specifications and methods of test. In the seven years of the committee's existence an ASTM Manual on Quality Control of Materials has been prepared; Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 – 50) have been prepared and adopted as standard by the Society; Symposiums on the Usefulness and Limitations of Samples, and on Bulk Sampling were held; and assistance has been given to various committees in the application of statistical methods to the preparation of standards.

Further to promote the purposes of the committee each ASTM technical committee will be invited to be represented on Committee E-11 by an associate member. The individual would be skilled in the application of statistical methods to standardization work and competent in the field of the technical committee. The preferred individual would be the chairman of a subcommittee on statistical methods or presentation of data. It is expected that this liaison will result in bringing to the attention of Committee E-11 technica committee problems involving the application of statistical methods. In turn the activities of Committee E-11 will be relayed back to the technical committees.

Active Task Groups of Committee E-11 are as follows:

No. 4 ASTM Sampling Plans
No. 5 Planning Interlaboratory Test Programs
No. 6 Number of Tests for a Desired Precision of An Average
No. 7 Bulk Sampling
No. 8 Smoothing Empirical Data
No. 9 Precision and Accuracy
No. 10 Design of Experiments

Recommended practices on several of the above subjects are currently in preparation.

This report has been submitted to letter ballot of the committee, which consists of 21 members; 18 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. Dodge,
Chairman.

O. P. Beckwith,
Secretary.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28-July 3, 1953.

REPORT OF COMMITTEE E-12

ON

APPEARANCE

Committee E-12 on Appearance held two meetings during the year: the first at New York, N. Y., on June 25, 1952, and the second at Detroit, Mich., on March 3 and 4, 1953.

NEW TENTATIVE

The proposed Tentative Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry, appended hereto,[1] is recommended for publication as tentative. This method has been developed in cooperation with Committees C-22 on Porcelain Enamel, D-1 on Paint, Varnish, Lacquer, and Related Products, and D-6 on Paper and Paper Products. It represents a proposed consolidation of a proposed method prepared by Committee C-22 and of the present Standard Method of Test for Daylight 45-deg, 0-deg Luminous Directional Reflectance of Paint Finishes (D 771 – 47), and the Standard Method of Test for 45-deg, 0-deg Directional Reflectance for Blue Light (Brightness) of Paper (D 985 – 50).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions (R. S. Hunter, chairman) has reached agreement on three definitions relating to gloss. These have been circulated for comment and suggestions, with general agreement having been obtained. The definitions are as follows:

Gloss.—The characteristics of an object which can produce shiny or lustrous appearance.

Luminous Fractional Reflectance.—The ratio of the luminous flux reflected to that incident on a specimen, for specified solid angles.

Specular Gloss.—The luminous fractional reflectance of a specimen in the direction of specular reflection.

Subcommittee II on Color and Spectral Characteristics (Dorothy Nickerson, chairman).—Group 1 of this subcommittee, under the chairmanship of Harry K. Hammond, prepared the proposed Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry, submitted with this report.

Group 4, under the chairmanship of Deane B. Judd, has indicated that at the meeting of the committee in June a consolidated test method for colorimetric specification from spectrophotometric measurements will be presented to the subcommittee.

The papers presented at the 1952 Symposium on Color Difference Specification have been published as a special technical publication.[2]

The advisability of setting up a new group to study the psychological aspects of color and color difference has been considered. The first project would be the visual evaluation of the color series being prepared in Subcommittee X on Optical Properties of Committee D-1 for instrumental evaluation of small color differences.

Subcommittee III on Gloss and Geometric Characteristics (H. K. Ham-

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[*] Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The new tentative was accepted by the Society and appears in the 1953 Supplement to Book of ASTM Standards, Part 4.

mond, III, chairman).—The major effort of this subcommittee concerned the organization and sponsorship of the Symposium on Gloss held in Detroit, Mich., on March 4, 1953, which reviewed and discussed the basic principles and concepts of gloss perception and measurement. The program included:

"Surfaces as Seen and Photographed," by Ralph M. Evans.

"The Physics of Surface Reflection," by Harry K. Hammond, III, and Daniel Smith.

"Gloss and its Measurement," by Richard S. Hunter.[2]

Moderator of Discussion—Deane B. Judd.

This program was summarized in the April, 1953, issue of the ASTM BULLETIN.

Subcommittee IV on Pictorial Representation (R. M. Evans, chairman) has been fortunate in obtaining the active interest of Ralph Evans as its chairman. Consideration is being given to the application of basic pictorial principles to the requirements of the technical committees of the Society.

This report has been submitted to letter ballot of the committee, which consists of 64 members; 43 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

M. REA PAUL,
Chairman.

DANIEL SMITH,
Secretary.

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[2] Published in ASTM BULLETIN, No. 186, December, 952, p. 48 (TP190).

REPORT OF COMMITTEE E-14
ON
MASS SPECTROMETRY*

Committee E-14 on Mass Spectrometry held one meeting during the past year, in Pittsburgh, Pa., on March 3, 1953.

A symposium on mass spectrometry was sponsored by the committee and was held in conjunction with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March 2 to 6, in Pittsburgh, Pa. The following 26 papers covering a large cross-section of the field of mass spectrometry were presented:

The Detection of Atoms and Free Radicals in Flames by Mass Spectrometric Techniques—S. N. Foner

Some Ions of High Kinetic Energy in Mass Spectra of Polyatomic Molecules—Fred L. Mohler

The Formation of Negative Ions of Sulfur Hexafluoride—A. J. Ahearn and N. B. Hannay

Half-life of Negative Metastable Ions—B. L. Donnally and H. E. Carr

Ionization and Dissociation by Electron Impact: 1-Butanethiol, 2-Butanethiol, and 3-Methyl-2-Propanethiol—G. L. Cook and J. J. Walsh

Mass Spectrometer Investigation of Higher Valence Uranium Compounds in UF—Russell Baldock, John R. Sites, L. O. Gilpatrick, and H. E. Carr

The Metropolitan-Vickers Mass Spectrometers —J. Blears

Accuracy and Reproducibility of Hydrocarbon Analysis in Routine Plant Control with a Metropolitan-Vickers Mass Spectrometer—D. L. Nicholson, M. Jones, and B. W. Bradford

A New Monitoring Mass Spectrometer—Gardner Wilson and A. P. Gifford

A Method for Determination of Values for Nitrous Oxide in Blood with the Mass Spectrometer—Franklin K. Dietzler, John Saari, Albert Faulconer, and Edward J. Baldes

Analysis of Mixtures of the Oxides of Nitrogen and Carbon—H. J. Frey and G. E. Moore

Mass Spectrometric Analysis of Mixtures Containing Nitric Oxide and Dinitrogen Tetroxide—R. A. Friedel, A. G. Sharkey, Jr., Janet L. Shultz, and C. R. Humbert

Mass Spectrometry of Solids—E. J. Serfass

Mass Spectrometric Study of Germanium—R. E. Honig

The Mass Spectrum of Lithium Iodide—Lewis Friedman

High-Temperature Mass Spectrometer Oven—J. B. Walton

Mass Dependence of the Gain of an Electron Multiplier from Observations on K-39, Na-23, W-184, and U-238—F. A. White and T. L. Collins

A Reliable Method of Counting Individual Positive Ions in a Mass Spectrometer for Beam Current of Less Than 10^{-15} Ampere—F. A. White

Lubricating Oil Analysis by Thermal Diffusion and Mass Spectrometry—F. W. Melpolder, R. A. Brown, T. A. Washall, W. Doherty, and W. S. Young

Analysis of Virgin Petroleum Naphthas by Mass and Infrared Spectroscopy—B. W. Thomas, J. A. Anderson, Jr., Annelle Elliott, H. E. Lumpkin, and R. B. Williams

Gain Control of Mass-Spectrometer Amplifier—S. Meyerson, H. M. Grubb, and W. H. Moeller

Computed Calibration Data for Gas Analysis by Mass Spectrometer—S. Meyerson

Structural Qualitative Analysis from Mass Spectra—J. M. McCrea

The Identification and Determination of Volatile Atmospheric Pollutants—Fred W. McLafferty, Gerald E. Clock, and Roland S. Gohlke

Mass Spectrometric Analysis of Six-and Seven-Carbon Alcohols—V. A. Yarborough

Mass Spectroscopic Studies of Mixtures of Water and Deuterium Oxide—John L. Warneck

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* Presented at the Fifth-sixth Annual Meeting of the Society, June 28-July 3, 1953.

A round-table discussion of high molecular weight mass spectrometry, a promising, relatively new development in the field, was sponsored by the committee, and also held in conjunction with the Pittsburgh meeting.

During the past year the following five subcommittees of Committee E-14 were organized:

Subcommittee I on Programs and Papers, W. J. Priestley, Jr., chairman;
Subcommittee II on Theoretical and Fundamental Aspects, F. L. Mohler, chairman;
Subcommittee III on High Molecular Weight and Solids Techniques, M. J. O'Neal, chairman;
Subcommittee IV on Methods, Data, and Nomenclature, H. M. Kelley, chairman; and
Subcommittee V on New Instruments and Techniques, H. F. Wiley, chairman.

Meetings are planned for September, 1953, in Chicago, in conjunction with meetings of the American Chemical Society, and for May, 1954, in New Orleans. Papers sessions on mass spectrometry are being planned for the New Orleans meeting.

Respectfully submitted on behalf of the committee,

W. S. YOUNG,
Chairman.

J. G. HUTTON,
Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

AN EXCURSION IN PETROLEUM CHEMISTRY[1]

TWENTY-SEVENTH EDGAR MARBURG LECTURE

BY FREDERICK D. ROSSINI[2]

I feel highly honored to have been invited to give the Edgar Marburg Lecture for 1953 before the American Society for Testing Materials. In keeping with the purpose of the series of Marburg Lectures, to describe developments in the promotion of knowledge of engineering materials, I thought it fitting to discuss petroleum, because researches in this field have occupied my attention for 20 years. Today's lecture, then, is an excursion in petroleum chemistry.

There is romance in petroleum chemistry research. The spectacular and vital accomplishments of the petroleum industry in World War II, in supplying for our country's defense enormous quantities of toluene for explosives, components for synthetic rubbers, and high-powered aviation fuel, would have been thought humanly impossible one or two decades earlier. Such developments are rooted in the research performed in the laboratories of the petroleum companies and in the fundamental research projects supported cooperatively by the petroleum industry through the American Petroleum Institute.[3]

Petroleum, one of our most important natural resources, is a very interesting and intriguing chemical substance. Although it contains small amounts of sulfur, nitrogen, and oxygen compounds, petroleum is largely hydrocarbon material, with molecules ranging in size from 1 to 50 or more carbon atoms and comprising a diversity of molecular types, as paraffins, cycloparaffins (or naphthenes), and aromatics (including benzene, toluene, naphthalene, and related compounds) (**8**).[4] Because petroleum contains many hydrocarbon compounds which are of themselves important chemicals of commerce, and because chemicals derived from petroleum are increasing greatly in number and importance, petroleum has come to be called Nature's storehouse of chemicals.

Going back into history, we find that early man long wondered about the thick dark liquid that in certain lands oozed from the earth or floated on natural waters (**9**). He learned many centuries ago that this strange substance would burn and give out great heat and light. Petroleum, or some portion thereof, as bitumen or asphalt, is mentioned in the Bible. History records that asphalt was used by man as early as 3800 B.C. (**4**).

Petroleum was found as an oily liquid

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[1] Presented at the Fifty-sixth Annual Meeting of the Society, July 1, 1953, Atlantic City, N. J.

[2] Silliman Professor, Head of the Department of Chemistry, and Director of the Petroleum Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.

[3] For reports on the work of the several research projects currently sponsored by the American Petroleum Institute, through its Advisory Committee on Fundamental Research on the Composition and Properties of Petroleum, the reader is referred to references **1, 2, 3, 4, 5, 6, 7.**

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 575.

exuding from the surface of the earth in tar springs, or on shores of lakes, or rising from springs beneath the beds of rivers. On the surface of water, petroleum would appear as a thin film, with rainbow colors, or in the form of dark globules that could be separated mechanically. In these forms, and in these limited quantities, petroleum was found in many places on earth—notably Rumania, Persia (now Iran), Italy, Trinidad, Cuba, India, and later the United States. In Western Pennsylvania, an enterprising business man by the name of Kier put up petroleum in bottles, labeled Kier's Rock Oil, and sold it as a medicine for the cure of many human ills.

In 1853, a young lawyer of New York City returned on a visit to his old home at Hanover, N. H. (10). There in the office of the Department of Chemistry of Dartmouth College, he saw, and was greatly impressed by, a small bottle of petroleum taken from an oil spring near Titusville in northwestern Pennsylvania. He discussed the commercial possibilities of petroleum with his law partner, who also was greatly impressed. They interested some capitalists of New Haven, Conn., with a view to organizing a company, buying the Pennsylvania farm where the oil springs were located, and marketing the petroleum as an article of commerce. But before making any investment, the New Haven capitalists thought that they should have an analysis made of the oil to determine its utility and value. For this purpose, the lawyer promoters engaged one of the most distinguished scientists of the country, Prof. Benjamin Silliman, Jr., of Yale College (10).

In the meantime, the two lawyers proceeded to Titusville, inspected the oil springs, purchased the farm on which they were located, and in December, 1854, organized the world's first petroleum company, called the Pennsylvania Rock Oil Company of New York. However, it was not easy to sell stock in the new venture, because the prospective investors knew little or nothing about petroleum, its uses, or potential commercial value (10).

But Professor Silliman soon answered these questions. In April, 1855, Silliman's completed report was placed in the hands of a friend in New York City, with instructions, however, not to deliver the report to the lawyer promoters until satisfactory arrangements were made by them to pay Silliman for his services in the amount of $526.08. The men scraped together enough money to pay the bill and secure the report. Within a short time, printed copies of the first scientific analysis of petroleum were ready for distribution.[5] Silliman's report was the turning point, for it dispelled doubts and encouraged investors in the venture to market the rock oil from the farm near Titusville.

I should like to read a few excerpts from Silliman's report (10). The cover page reads:

* * *

REPORT
on the
ROCK OIL, OR PETROLEUM
from
VENANGO CO., PENNSYLVANIA,
WITH SPECIAL REFERENCE TO ITS USE FOR
ILLUMINATION AND OTHER PURPOSES
BY B. SILLIMAN, JR.,
Prof. of General and Applied Chemistry,
Yale College

* * *

The report itself begins as follows (10):

Messrs. Eveleth, Bissell and Reed,
Gentlemen,—

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[5] For additional historical material, the reader is referred to references **13**, **14**, **15**. In particular, reference **15** deals with the publication in 1833 of an article by Benjamin Silliman, Sr., entitled "Notice of a Fountain of Petroleum Called the Oil Spring," in which the elder Silliman describes certain tests he made on petroleum from an oil spring.

> I herewith offer you the results of my somewhat extended researches upon the Rock Oil, or Petroleum, from Venango County, Pennsylvania, which you have requested me to examine with reference to its value for economical purposes.

On page 5 of the report, we read (10):

> The Crude oil, as is gathered on your lands, has a dark brown color, which, by reflected light, is greenish or bluish. It is thick even in warm weather—about as thick as thin molasses. In very cold weather it is somewhat more stiff, but can always be poured from a bottle even at 15 F below zero. Its odor is strong and peculiar, and recalls to those who are familiar with it, the smell of Bitumen and Naphtha. Exposed for a long time to the air, it does not thicken or form a skin on its surface, and, in no sense, can it be called a drying oil. The density of the Crude oil is 882, water being 1000. It boils only at a very high temperature, and yet it begins to give off a vapor at a temperature not greatly above that of boiling water. It takes fire with some difficulty, and burns with an abundant smoky flame. It stains paper with the appearance of ordinary fat oils, and feels smooth and greasy between the fingers. It is frequently used in its crude state to lubricate coarse machinery. In chemical characters (*properties*), it is entirely unlike the fat oils. Most of these characters (*properties*) are common to Petroleum from various places.

The conclusion of Silliman's report is interesting to read (10):

> In conclusion, gentlemen, it appears to me that there is much ground for encouragement in the belief that your Company have in their possession a raw material from which, by simple and not expensive process, they may manufacture very valuable products.
>
> It is worthy of note that my experiments prove that nearly the *whole* of the raw product may be manufactured without waste, and this solely by a well directed process which is in practice one of the most simple of all chemical processes.
>
> There are suggestions of a practical nature, as to the economy of your manufacture, when you are ready to begin operations, which I shall be happy to make, should the Company require it—meanwhile, I remain, gentlemen,
>
> Your ob't serv't,
>
> B. SILLIMAN, Jr.,
> Prof. of Chemistry in Yale College.
>
> New Haven, April 16, 1855.

Four years later, in 1859, the famous Drake well was completed, near Titusville, Pa., to become the first petroleum well in history. This well, drilled to a depth of 69½ ft, produced about 25 barrels of crude oil per day—in those days an extremely large production. But we all know how the production of petroleum in the continental United States since then has grown to astonishing proportions, as indicated by the following figures, in millions of barrels per year: 0.002 in 1860; 26 in 1880; 64 in 1900; 443 in 1920; 1300 in 1940; and 2200 in 1952. Since 1880, the United States production has been about 70 per cent of the world production.

Notwithstanding the enormous production, the proved reserves of crude oil in the United States have also increased to large proportions, being as follows, in billions of barrels: 3 in 1900; 7 in 1920; 19 in 1940; and 28 in 1952. For the continental United States, the ratio of proved reserves to the annual production has decreased somewhat over the years, being 21 in 1910, 16 in 1920, 15 in 1930, 14 in 1940, and 13 in 1950. It is seen from these figures that the ability of man to locate and recover petroleum is keeping reasonable pace with his present needs.

Prior to about 1900, the refining of petroleum consisted essentially in using the process of simple distillation to separate crude oil into the fractions we now identify as gasoline, kerosine, gas-oil, and lubricating oil. Most of the early commercial use was of the kerosine for illumination, and of the lubricating oil for lubrication. In those early days, the gasoline fraction was a drug on the market and, because of its volatility, its physical disposal posed a real and hazardous problem. After about 1900, however, more and more of the gasoline was needed to operate the increasingly great number of automobiles appearing on the American highways. Soon there even became a shortage of gasoline in terms of the quantity of it which could be obtained from a barrel of crude oil. Fortunately, the development and introduction of the thermal cracking process by the petroleum industry about the time of World War I served to increase the quantity of gasoline and at the same time to dispose of the surplus gas-oil by the thermal cracking of the latter into the much needed gasoline.

After the end of World War I, the petroleum industry began to be seriously concerned about the chemical constitution of its raw material. It was quite clear that increasing fundamental knowledge of the mixture of chemical compounds constituting petroleum would lead not only to a more economical use of petroleum in the existing processes, but also would lead to the production from petroleum of entirely new substances for the needs of man.

In 1925, the petroleum industry, through the American Petroleum Institute (API), embarked on a program of fundamental research on the origin, recovery, production, composition, and properties of petroleum. The purpose of this program was to obtain fundamental, basic, and background information concerning petroleum which would benefit the industry as a whole in fields of broad, but primarily noncompetitive, interest and would not be directly applicable to industrial operations or processes (**1**).

Among the many API research projects that were carried on under this program, we single out for discussion here the two with which your speaker has been connected, namely, the API Research Project 6, on "Analysis, Purification, and Properties of Petroleum Hydrocarbons," established in 1927 and involving to date about 350 man-years of work, and the API Research Project 44, on "Data on Hydrocarbons and Related Compounds," established in 1942 and involving to date about 85 man-years of work.[6]

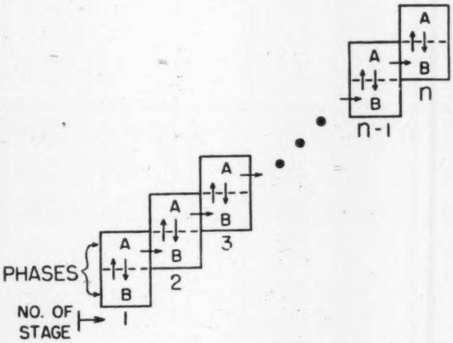

FIG. 1.—Schematic Diagram of a Simple Fractionating Process of *n* Stages, Involving Thermodynamic Equilibrium of Two Components in Each of Two Phases.

From Rossini (**11**).

API RESEARCH PROJECT 6

The API Research Project 6 carries on the following investigations:[7]

1. Fractionation, analysis, and isolation of hydrocarbons from petroleum. The general problem set before the Project was to start with a large quantity of a selected original crude petroleum known to have intermediate properties and by the appropriate interlocking of suitable physical methods of fractionation resolve the petroleum into its major constituent compounds.

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[6] A summarizing description of the work of the API Research Project 6 is given in reference **2** and of the API Research Project 44 in reference **4**.

[7] For a detailed account of the work of the API Research Project 6, the reader is referred to reference **12**.

2. Development and operation of fractionating processes. In order to separate the petroleum into its components, fractionating processes of high separating power are developed and operated.

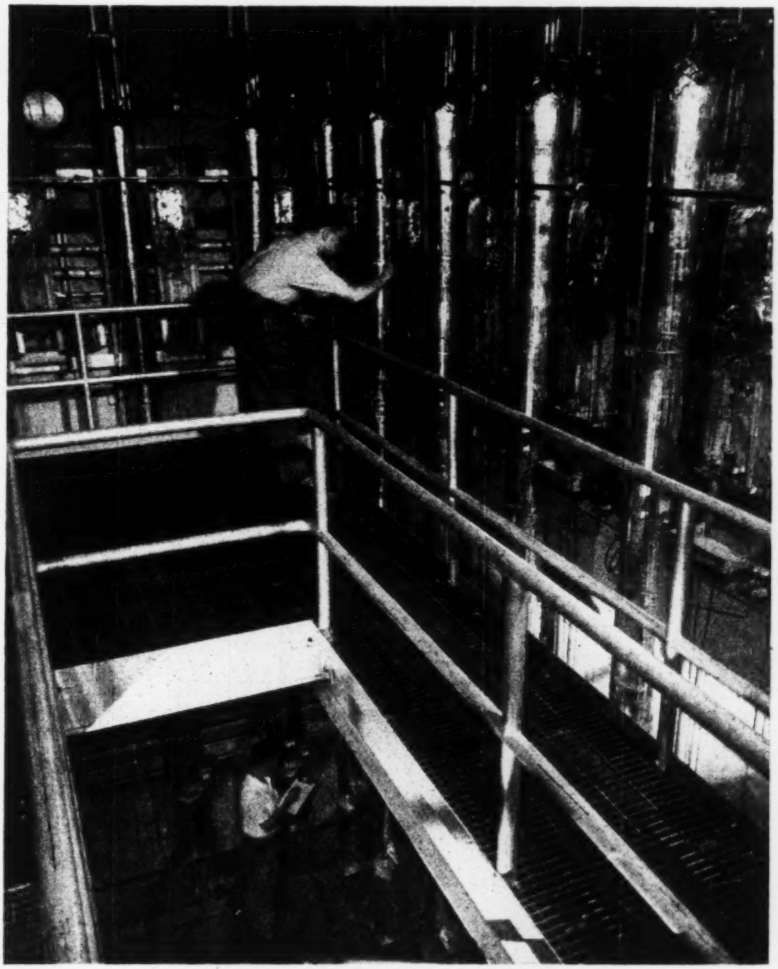

FIG. 2.—Nature Did Not Make Petroleum Simple.

Petroleum is one of the most complex mixtures of nature. The tall distilling columns shown here in the laboratories of the API Research Project 6 at the Carnegie Institute of Technology operate continuously 24 hr per day, 7 days per week, with fractionating efficiency equivalent to 200 theoretical plates, unlocking the secrets of the composition of the raw material of the petroleum industry.

3. Purification of API Standard and API Research hydrocarbons. To identify compounds isolated from petroleum, synthetic API Research hydrocarbons are purified for reference and research. To satisfy the needs of the industry for standard hydrocarbons for calibrating spectrometers, API Standard hydrocarbons are purified.

4. Measurement of physical properties of API Research hydrocarbons. For purposes of record and identification, accurate measurements are made of the simple physical properties of the highly purified API Research hydrocarbons.

TABLE I.—CHARACTERISTICS OF THE 13 REGULAR PACKED DISTILLING COLUMNS OF API RESEARCH PROJECT 6 AS OF JUNE 30, 1953.

	Rectifying Section				Pot		Minimum Operating Residue (approximate), ml	Operating Pressure Drop,[c] mm Hg	Operating Throughput,[c] ml per hr	Rate of Collection of Product,[c] ml per hr	Operating Reflux Ratio (approximate)	Estimated Number of Theoretical Plates (at total reflux)	Required Time for Distillation of Maximum Charge[d] (approximate), hr
	Diameter, mm	Length[a]		Packing[b]	Actual Volume, liters	Maximum Charging Capacity, liters							
		m	ft										
1.....	11	2.74	9	HG	3.2	2.8	40	12	300	2.0	150	150	1420
2.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
3.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
4.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
5.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
6.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
7.....	16	3.66	12	HG	7.2	6.0	100	20	800	4.0	200	200	1515
8.....	22	3.66	12	HG	7.2	6.0	100	20	1400	7.0	200	200	885
9.....	22	3.66	12	HG	7.2	6.0	100	20	1400	7.0	200	200	885
10.....	25	3.66	12	HG	7.2	6.0	100	20	1800	9.0	200	200	695
11.....	25	4.57	15	SSH	7.2	6.0	175	26	1600	11.0	150	150	570
12.....	25	4.57	15	SSH	18.0	15.0	300	26	1600	11.0	150	150	1375
13.....	25	4.57	15	SSH	18.0	15.0	300	26	1600	11.0	150	150	1375

[a] This is the actual length of packed section. The total length of each column, from the bottom of the pot to the end of the condenser, is approximately 6.9 m (20 ft).

[b] SSH represents stainless steel helices, 3/32 in. in diameter, made of No. 30 AWG wire; HG represents Heli-Grid packing.

[c] The values listed for pressure drop, throughput, and rate of collection of product refer to operations at 760 mm mercury. For operations at lower pressures, values for the throughput and rate of collection of product are lower.

[d] Including the initial hours for attainment of equilibrium. For distillations at 30 mm mercury, approximately 4000 hr are required for the distillation of the maximum charge in columns 12 and 13.

The methods of fractionation used in the work on the API Research Project 6 include the following:

1. Distillation (involving a liquid phase and a gas phase).
   (*a*) Regular, at one pressure (at 30, 55, 214, and 760 mm mercury).
   (*b*) Alternately at different pressures (at 30, 55, 214, and 760 mm mercury).
   (*c*) At very low pressures (0.01 to 0.1 mm mercury).
2. Extraction (involving two liquid phases).
   (*a*) With one solvent phase.
   (*b*) With two solvent phases.
3. Adsorption (involving a liquid phase and a solid-adsorbed phase).
   (*a*) Regular.
   (*b*) With one or more added components.
4. Crystallization (involving a liquid phase and a solid phase).
   (*a*) Regular.
   (*b*) With selected solvent.

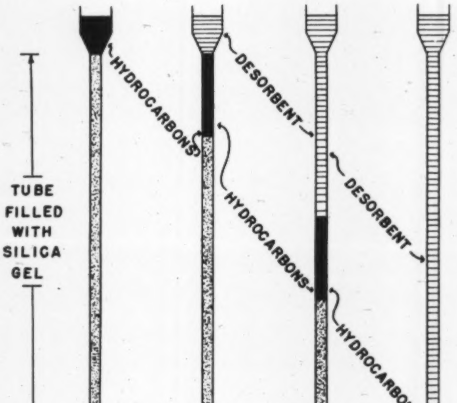

FIG. 3.—Schematic Diagram of the Simple Fractionating Process of Adsorption.

5. Formation of molecular or clathrate compounds.

Among the foregoing fractionating processes, separation by size of molecules is produced by distillation at one fixed pressure, whereas separation by type of molecules is produced by distillation alternately at different pressures, distillation azeotropically, extraction, and adsorption.

Figure 1 shows a schematic diagram of a simple fractionating process of *n* stages, involving thermodynamic equilibrium of two components in each of two phases (11). In the first stage, phase A is in equilibrium with phase B. Phase A of stage 1 has the same composition as phase B of stage 2. A new equilibrium exists between phases A and B in stage 2. Phase A of stage 2 has the same composition as phase B of stage 3. And so on through the *n* stages. The two phases involved may be any combination of gas, liquid, or solid, depending upon the particular process being used. One important point to emphasize is the following: if a given process produces in one stage any measureable separation, however small, the process can usually be made a very successful one simply by using a sufficiently large number of stages, *n*.

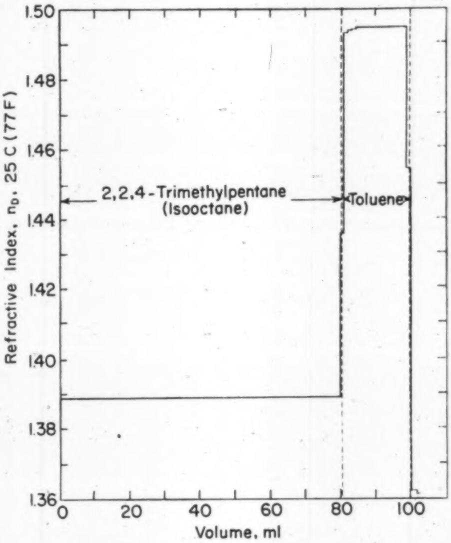

FIG. 4.—Separation by Adsorption of Toluene and 2,2,4-Trimethylpentane ("*Iso*octane").

Figure 2 is a photograph of the distillation laboratory of the API Research Project 6 (12). Normally, this laboratory operates continuously 24 hr per day, every day in the year. The laboratory has 13 complete distilling columns with the characteristics shown in Table I. One of the representative columns, such as Column 5, has a fractionating power of 200 equivalent theoretical plates, a charging capacity of 6 liters (about 1½ gal), a throughput of 800 ml per hr, a reflux ratio of 200 to 1, and a product rate of 4 ml per hr. This means that a full charge requires about 1500 hr or 2 months of continuous distillation at 760 mm mercury (12).

A schematic diagram of the simple fractionating process of adsorption is given in Fig. 3. The desorbent (ethyl alcohol) forces the zone of hydrocarbons (shown in solid black) down over the solid adsorbent (silica gel). As the zone of hydrocarbons passes down over the adsorbent, those hydrocarbons which are more strongly adsorbed lag behind while those which are less strongly adsorbed are pushed forward and are the first to issue from the bottom of the adsorption tube. In actual practice, we have apparatus of this kind ranging in size from pyrex glass tubes ⅜ in. in diameter and 3 ft in length up to stainless steel tubes 2 in. in diameter and 65 ft in length (12).

One of the small glass adsorption columns can be used to determine the amount of aromatic hydrocarbons in a sample of gasoline. The results of the separation by adsorption of a test mixture made up of 20 ml of toluene and 80 ml of 2,2,4-trimethylpentane ("*iso*octane") is shown in Fig. 4. The horizontal scale gives the volume of filtrate issuing from the apparatus, with the refractive index of the filtrate being plotted vertically. The separation is seen to be quite sharp. This apparatus and method became the basis of ASTM method of test for aromatic hydrocarbons in gasoline by adsorption (D 936),[8] developed under Section C on Analysis of Hydrocarbons by Adsorption of Research Division IV on Hydrocarbon Analysis of ASTM Committee D-2 on Petroleum Products and Lubricants.

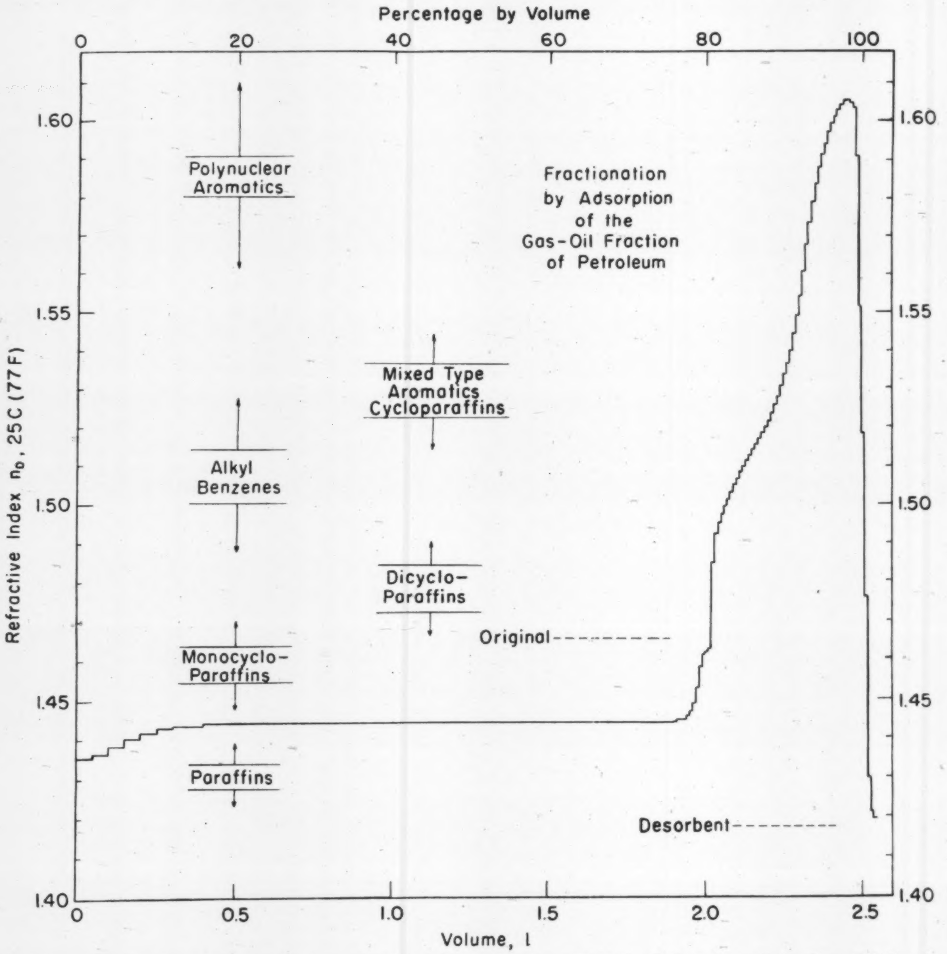

FIG. 5.—Results of the Fractionation by Adsorption in a Tall Steel Column of the Gas-Oil Fraction of Petroleum.

From Rossini, Mair, and Streiff (**12**).

Figure 5 illustrates how the gas-oil fraction of petroleum, which is between kerosine and lubricating oil, is separated by adsorption in a tall steel column. It is seen that this one-pass operation yields a powerful separation of this complex mixture into certain classes of components.

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[8] Tentative Method of Test for Aromatic Hydrocarbons in Olefin-Free Gasolines by Silica Gel Adsorption (D 936-51 T), 1952 Book of ASTM Standards, Part 5, p. 376.

FIG. 6.—Photograph of the Site of the Brett No. 6 Well, near Ponca City, Okla., on March 10, 1940. Frederick D. Rossini (Director, API Research Project 6) is at the left and Bert H. Lincoln (then Chief Chemist, Continental Oil Co.) at the right.

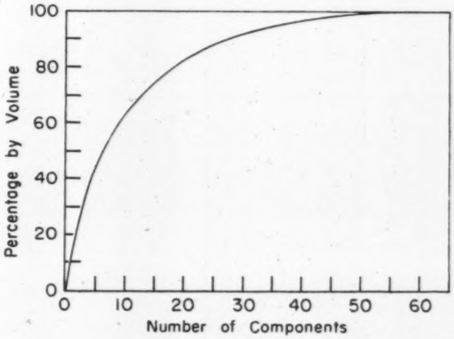

FIG. 7.—Relation Between the Number of Components in the Light Gasoline Fraction, 40 to 132 C, and the Percentage of the Material Constituted by Them.

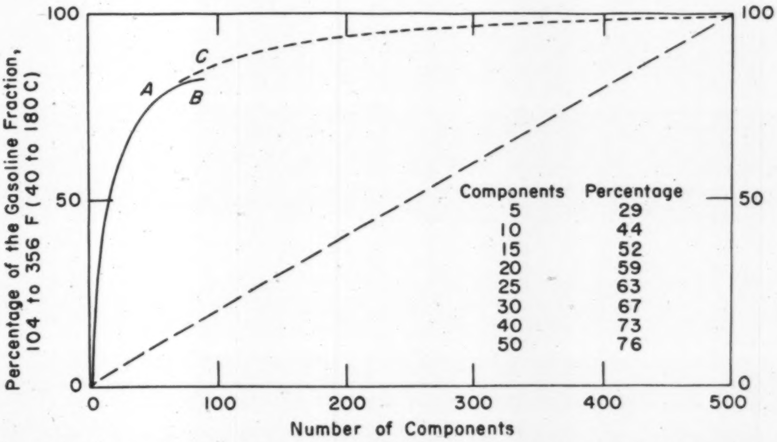

FIG. 8.—Relation Between the Number of Components in the Gasoline Fraction, 40 to 180 C, and the Percentage of the Material Constituted by Them.

Figure 6 shows the site of the Brett No. 6 well near Ponca City, Okla. This well was drilled in 1919, producing at a depth of 3870 ft; it was placed "on the pump" in 1926 and produced until 1936 when the well was plugged and abandoned because of salt water encroachment. In 1928, there was taken from this one well 600 gal of crude petroleum, which has been under investigation by the API Research Project 6 since then.

Table II gives a list of the 135 hydrocarbons isolated from the Project's representative petroleum as of June 30,

TABLE II.—HYDROCARBONS ISOLATED FROM ONE REPRESENTATIVE PETROLEUM.
By the API Research Project 6 as of June 30, 1953.

No.	Formula	Compound	Type[a]	Boiling Point at Atmosphere,[b] deg Cent	Purity of the Best Sample Isolated,[c] mole per cent	Estimated Amount in the Crude Petroleum,[d] volume per cent	References to Research Papers in Which the Work is Described[e]
1....	CH_4	Methane	Normal paraffin	−161.49	[f]	[f]	...
2....	C_2H_6	Ethane	Normal paraffin	−88.63	[f]	[f]	...
3....	C_3H_8	Propane	Normal paraffin	−42.07	[f]	[f]	...
4...	C_4H_{10}	Isobutane	Branched paraffin	−11.73	[f]	[f]	...
5....	C_4H_{10}	*n*-Butane	Normal paraffin	−0.50	[f]	[f]	...
6....	C_5H_{12}	2-Methylbutane	Branched paraffin	27.85	[f]	[f]	...
7....	C_5H_{12}	*n*-Pentane	Normal paraffin	36.07	[f]	[f]	...
8....	C_5H_{10}	Cyclopentane	Cyclopentane	49.26	[f]	0.05	84
9....	C_6H_{14}	2,2-Dimethylbutane	Branched paraffin	49.74	[f]	0.04	84
10....	C_6H_{14}	2,3-Dimethylbutane	Branched paraffin	57.99	97	0.08	8, 84
11....	C_6H_{14}	2-Methylpentane	Branched paraffin	60.27	99.97	0.37	8, 84, 97
12....	C_6H_{14}	3-Methylpentane	Branched paraffin	63.28	99.9	0.35	8, 84, 97
13....	C_6H_{14}	*n*-Hexane	Normal paraffin	68.74	99.9	1.80	12, 84, 97
14....	C_6H_{12}	Methylcyclopentane	Cyclopentane	71.81	98.7	0.87	14, 84
15....	C_7H_{16}	2,2-Dimethylpentane	Branched paraffin	79.20	97	0.02	21, 84
16....	C_6H_6	Benzene	Benzene	80.10	99.6	0.15	12, 84
17....	C_7H_{16}	2,4-Dimethylpentane	Branched paraffin	80.50	[f]	0.08	84
18....	C_6H_{12}	Cyclohexane	Cyclohexane	80.74	99.9	0.71	13, 84
19....	C_7H_{14}	1,1-Dimethylcyclopentane	Cyclopentane	87.85	98	0.16	26, 84
20....	C_7H_{16}	2,3-Dimethylpentane	Branched paraffin	89.78	58[h]	0.15[i]	84, 127
21....	C_7H_{16}	2-Methylhexane	Branched paraffin	90.05	97	0.73[i]	26, 84, 127
22....	C_7H_{14}	1,trans-3-Dimethylcyclopentane	Cyclopentane	90.77	85	0.87[i]	75, 84, 127
23....	C_7H_{14}	1,cis-3-Dimethylcyclopentane	Cyclopentane	91.72	51[h]	0.21[i]	127
24....	C_7H_{16}	3-Methylhexane	Branched paraffin	91.85	93	0.51	75, 84, 127
25....	C_7H_{14}	1,trans-2-Dimethylcyclopentane	Cyclopentane	91.87	93	0.48[i]	75, 84, 127
26....	C_7H_{16}	3-Ethylpentane	Branched paraffin	93.48	98.7[h]	0.06	127
27....	C_7H_{16}	*n*-Heptane	Normal paraffin	98.43	99.9	2.3	18, 84
28....	C_7H_{14}	Methylcyclohexane	Cyclohexane	100.93	99.8	1.6	18, 84
29....	C_7H_{14}	Ethylcyclopentane	Cyclopentane	103.47	98	0.16	105
30....	C_8H_{16}	1,1,3-Trimethylcyclopentane	Cyclopentane	104.89	98.1	0.30	105
31....	C_8H_{18}	2,2-Dimethylhexane	Branched paraffin	106.84	50[h]	0.01	105
32....	C_8H_{18}	2,5-Dimethylhexane	Branched paraffin	109.10	55[h]	0.06[h]	125
33....	C_8H_{16}	1,trans-2,cis-4-Trimethylcyclopentane	Cyclopentane	109.29	84[h]	0.22	125
34....	C_8H_{18}	2,4-Dimethylhexane	Branched paraffin	109.43	41[h]	0.06[h]	125
35....	C_8H_{18}	2,2,3-Trimethylpentane	Branched paraffin	109.84	1.0[h]	0.004[h]	125
36....	C_8H_{16}	1,trans-2,cis-3-Trimethylcyclopentane	Cyclopentane	110.2	98.6	0.26	125
37....	C_7H_8	Toluene	Benzene	110.62	98	0.51	10, 84
38....	C_8H_{18}	3,3-Dimethylhexane	Branched paraffin	111.97	86[h]	0.03	125
39....	C_8H_{18}	2,3,4-Trimethylpentane	Branched paraffin	113.47	17[h]	0.005[h]	125
40....	C_8H_{16}	1,1,2-Trimethylcyclopentane	Cyclopentane	113.73	98	0.06	125
41....	C_8H_{18}	2,3,3-Trimethylpentane	Branched paraffin	114.76	10[h]	0.006[h]	125
42....	C_8H_{18}	2,3-Dimethylhexane	Branched paraffin	115.61	65[h]	0.07[h]	125
43....	C_8H_{18}	2-Methyl-3-ethylpentane	Branched paraffin	115.65	51[i]	0.06[h]	125
44....	C_8H_{16}	1,cis-2,trans-4-Trimethylcyclopentane	Cyclopentane	116.73	84[i]	0.01[i]	[g]
45....	C_8H_{16}	1,cis-2,trans-3-Trimethylcyclopentane	Cyclopentane	117.5	90[i]	0.07[i]	[g]
46....	C_8H_{18}	2-Methylheptane	Branched paraffin	117.65	90	0.90[i]	27,[g]
47....	C_8H_{18}	4-Methylheptane	Branched paraffin	117.71	34[i]	0.20[i]	[g]
48....	C_8H_{18}	3,4-Dimethylhexane	Branched paraffin	117.72	40[i]	0.13[i]	[g]
49....	C_8H_{18}	3-Methyl-3-ethylpentane	Branched paraffin	118.26	6[i]	0.02[i]	[g]
50....	C_8H_{18}	3-Ethylhexane	Branched paraffin	118.53	43[i]	0.09[i]	[g]
51....	C_7H_{14}	Cycloheptane	Cycloheptane	118.79	90	0.01[i]	[g]
52....	C_8H_{18}	3-Methylheptane	Branched paraffin	118.92	98[i]	0.30[i]	[g]
53....	C_8H_{16}	1,trans-4-Dimethylcyclohexane	Cyclohexane	119.35	75[i]	0.25[i]	[g]
54....	C_8H_{16}	1,1-Dimethylcyclohexane	Cyclohexane	119.54	84[i]	0.06[i]	[g]
55....	C_8H_{16}	1,cis-3-Dimethylcyclohexane	Cyclohexane	120.09	84[i]	0.63[i]	42,[g]
56....	C_8H_{16}	1-Methyl-trans-3-ethylcyclopentane	Cyclopentane	120.8	57[i]	0.12[i]	[g]
57....	C_8H_{16}	1-Methyl-cis-3-ethylcyclopentane	Cyclopentane	121.4			
58....	C_8H_{16}	1-Methyl-trans-2-ethylcyclopentane	Cyclopentane	121.2	74[i]	0.14[i]	[g]
59....	C_8H_{16}	1-Methyl-1-ethylcyclopentane	Cyclopentane	121.52	65[i]	0.03[i]	[g]
60....	C_9H_{18}	1,1,cis-3,trans-4-Tetramethylcyclopentane	Cyclopentane	121.6	83[i]	0.04[i]	[g]
61....	C_8H_{16}	1,trans-2-Dimethylcyclohexane	Cyclohexane	123.42	98[i]	0.31[i]	48,[g]
62....	C_9H_{20}	2,2,5-Trimethylhexane	Branched paraffin	124.08	11[i]	0.002[i]	[g]
63....	C_8H_{16}	1,cis-4-Dimethylcyclohexane	Cyclohexane	124.32	76[i]	0.09[i]	[g]
64....	C_8H_{16}	1,trans-3-Dimethylcyclohexane	Cyclohexane	124.45	49[i]	0.07[i]	[g]

TABLE II.—*Continued.*

No.	Formula	Compound	Type[a]	Boiling Point at Atmosphere,[b] deg Cent	Purity of the Best Sample Isolated,[c] mole per cent	Estimated Amount in the Crude Petroleum,[d] volume per cent	References to Research Papers in Which the Work is Described[e]
65....	C_8H_{18}	*n*-Octane	Normal paraffin	125.66	99.2	1.9	11, [g]
66....	C_8H_{16}	Isopropylcyclopentane	Cyclopentane	126.42	15[i]	0.01[i]	[g]
67....	C_9H_{18}	Tetramethylcyclopentane[m]	Cyclopentane	127.4	90	0.11	[g]
68....	C_8H_{16}	1-Methyl-cis-2-ethylcyclopentane	Cyclopentane	128.05	52[i]	0.04[i]	[g]
69....	C_8H_{16}	1,cis-2-Dimethylcyclohexane	Cyclohexane	129.73	45[i]	0.06[i]	[g]
70....	C_8H_{16}	*n*-Propylcyclopentane	Cyclopentane	130.95	49[i]	0.06[i]	[g]
71....	C_9H_{20}	2,3,5-Trimethylhexane	Branched paraffin	131.34	16[i]	0.03[i]	[g]
72....	C_8H_{16}	Ethylcyclohexane	Cyclohexane	131.78	94	0.37[i]	44,[g]
73....	C_9H_{20}	2,6-Dimethylheptane	Branched paraffin	135.21	98.6	0.05	52, 70
74....	C_8H_{10}	Ethylbenzene	Benzene	136.19	96	0.19	28, 84
75....	C_9H_{18}	1,1,3-Trimethylcyclohexane	Cyclohexane	136.63	99.9	0.2	37
76....	C_8H_{10}	*p*-Xylene	Benzene	138.35	99.8	0.10	24, 84
77....	C_8H_{10}	*m*-Xylene	Benzene	139.10	99.9	0.51	24, 84
78....	C_9H_{20}	2,3-Dimethylheptane	Branched paraffin	140.5	60	0.05	68
79....	C_9H_{18}	Trimethylcyclohexane[m]	Cyclohexane	141.2	95	0.2	68
80....	C_9H_{20}	4-Methyloctane	Branched paraffin	142.48	80	0.1	56
81....	C_9H_{20}	2-Methyloctane	Branched paraffin	143.26	99.9	0.4	56
82....	C_9H_{20}	3-Methyloctane	Branched paraffin	144.18	95	0.1	56
83....	C_8H_{10}	*o*-Xylene	Benzene	144.41	99.7	0.27	24, 84
84....	C_9H_{18}	Monocycloparaffin[m]	Monocycloparaffin	145.6	99	[f]	[g]
85....	C_9H_{16}	Dicycloparaffin[m]	Dicycloparaffin	146.7	99	[f]	[g]
86....	C_9H_{20}	*n*-Nonane	Normal paraffin	150.80	99.94	1.8	17, 103, [g]
87....	C_9H_{12}	Isopropylbenzene	Benzene	152.39	99.8	0.07[j]	61, 84, 111
88....	C_9H_{12}	*n*-Propylbenzene	Benzene	159.22	98	0.09[j]	84, 111, [g]
89....	C_9H_{12}	1-Methyl-3-ethylbenzene	Benzene	161.30	99[h]	0.17[j]	111, [g]
90....	C_9H_{12}	1-Methyl-4-ethylbenzene	Benzene	161.99	94[h]	0.06[j]	111, [g]
91....	C_9H_{12}	1,3,5-Trimethylbenzene	Benzene	164.72	99.9	0.12[j]	33, 111
92....	C_9H_{12}	1-Methyl-2-ethylbenzene	Benzene	165.15	89[h]	0.09[j]	111, [g]
93....	$C_{10}H_{22}$	4-Methylnonane	Branched paraffin	165.7	96	0.1	[g]
94....	$C_{10}H_{22}$	2-Methylnonane	Branched paraffin	166.8	99.9	0.3	[g]
95....	$C_{10}H_{22}$	3-Methylnonane	Branched paraffin	167.8	98	0.1	[g]
96....	$C_{10}H_{14}$	tert-Butylbenzene	Benzene	169.12	[f]	0.01[j]	111
97....	C_9H_{12}	1,2,4-Trimethylbenzene	Benzene	169.35	99.7	0.51[j]	33, 97, 111
98....	$C_{10}H_{14}$	Isobutylbenzene	Benzene	172.76	[f]	[f]	[g]
99....	$C_{10}H_{14}$	sec-Butylbenzene	Benzene	173.30	[f]	[f]	[g]
100....	$C_{10}H_{22}$	*n*-Decane	Normal paraffin	174.12	99.9	1.8	19, [g]
101....	C_9H_{12}	1,2,3-Trimethylbenzene	Benzene	176.08	99.8	0.12	33, 97
102....	$C_{10}H_{14}$	1-Methyl-3-propylbenzene	Benzene	181.80	[f]	[f]	[g]
103....	$C_{10}H_{14}$	1,2-Diethylbenzene	Benzene	183.42	[f]	[f]	[g]
104....	$C_{10}H_{14}$	1-Methyl-2-Propylbenzene	Benzene	184.80	[f]	[f]	[g]
105....	$C_{10}H_{14}$	1,4-Dimethyl-2-ethylbenzene	Benzene	186.91	[f]	[f]	[g]
106....	$C_{10}H_{18}$	trans-Decahydronaphthalene	Dicycloparaffin	187.25	[f]	[f]	[g]
107....	$C_{10}H_{14}$	1,3-Di methyl-4-ethylbenzene	Benzene	188.41	[f]	[f]	[g]
108....	$C_{10}H_{14}$	1,2-Dimethyl-3-ethylbenzene	Benzene	193.91	[f]	[f]	[g]
109....	$C_{11}H_{24}$	*n*-Undecane	Normal paraffin	195.89	99.97	1.6	116,
110....	$C_{10}H_{14}$	1,2,4,5-Tetramethylbenzene	Benzene	196.80	[f]	[f]	[g]
111....	$C_{10}H_{14}$	1,2,3,5-Tetramethylbenzene	Benzene	198.00	[f]	[f]	[g]
112....	$C_{11}H_{20}$	Dicycloparaffin	Dicycloparaffin	202.5	[f]	[f]	[g]
113....	$C_{11}H_{16}$	Alkyl benzene[m]	Benzene	204.1	98	0.06	[g]
114....	$C_{10}H_{12}$	4-Methylindan	Aromatic-cycloparaffin	204.5	68[i]	[f]	[g]
115....	$C_{10}H_{14}$	1,2,3,4-Tetramethylbenzene	Benzene	205.04	99.9	0.2	79
116....	$C_{11}H_{16}$	1,3-Dimethyl-4-*n*-propylbenzene[n]	Benzene	206.6	96	0.03	[g]
117....	$C_{10}H_{12}$	1,2,3,4-Tetrahydronaphthalene	Aromatic-cycloparaffin	207.57	98.5	0.03	79
118....	$C_{11}H_{16}$	1,2-Dimethyl-4-*n*-propylbenzene[n]	Benzene	208.5	99	0.03	[g]
119....	$C_{11}H_{16}$	Trimethylethylbenzene[m]	Benzene	212.3	97	0.04	[g]
120....	$C_{12}H_{26}$	*n*-Dodecane	Normal paraffin	216.28	99.9	1.4	74
121....	$C_{10}H_8$	Naphthalene	Dinuclear aromatic	217.96	99.9	0.06	74
122....	$C_{11}H_{14}$	Aromatic-cycloparaffin[m]	Aromatic-cycloparaffin	220.7	97	0.04	[g]
123....	$C_{11}H_{14}$	6-Methyl-[1,2,3,4-tetrahydronaphthalene]	Aromatic-cycloparaffin	229.03	99.5	0.09	79
124....	$C_{11}H_{14}$	5-Methyl-[1,2,3,4-tetrahydronaphthalene]	Aromatic-cycloparaffin	234.35	99.7	0.08	79
125....	$C_{13}H_{28}$	*n*-Tridecane	Normal paraffin	235.44	98	1.2	[g]
126....	$C_{11}H_{10}$	2-Methylnaphthalene	Dinuclear aromatic	241.05	99.9	0.2	74
127....	$C_{11}H_{10}$	1-Methylnaphthalene	Dinuclear aromatic	244.64	99.7	0.1	74
128....	$C_{14}H_{30}$	*n*-Tetradecane	Normal paraffin	253.57	98.5[k]	1.0	[g]
129....	$C_{12}H_{10}$	Biphenyl	Dinuclear aromatic	255.2	[f]	[f]	[g]
130....	$C_{12}H_{12}$	2,6-Dimethylnaphthalene	Dinuclear aromatic	262	[f]	[f]	[g]
131....	$C_{12}H_{12}$	Dimethylnaphthalene[m]	Dinuclear aromatic	268	[f]	[f]	[g]

(Continued on page 556)

TABLE II.—*Concluded.*

No.	Formula	Compound	Type[a]	Boiling Point at Atmosphere,[b] deg Cent	Purity of the Best Sample Isolated,[c] mole per cent	Estimated Amount in the Crude Petroleum,[d] volume per cent	References to Research Papers in Which the Work is Described[e]
132....	$C_{15}H_{32}$	*n*-Pentadecane	Normal paraffin	270.63	98.5[k]	0.8	[g]
133....	$C_{13}H_{14}$	Trimethylnaphthalene[m]	Dinuclear aromatic	285	[f]	[f]	[g]
134....	$C_{16}H_{34}$	*n*-Hexadecane	Normal paraffin	286.79	98[k]	0.7	[g]
135....	$C_{17}H_{36}$	*n*-Heptadecane	Normal paraffin	301.82	97[k]	0.6	[g]

[a] The compounds are classified according to the following types: normal paraffin; branched paraffin; cyclopentane (and its alkyl derivatives); cyclohexane (and its alkyl derivatives); benzene (and its alkyl derivatives); dicycloparaffin; dinuclear aromatic; aromatic-cycloparaffin (mixed type). "Monocycloparaffin" indicates either the "cyclopentane" or the "cyclohexane" type.

[b] This is the value for the pure compound, as taken from the tables and files of the American Petroleum Institute Research Project 44, and is not necessarily the temperature at which the compound appears in the distillation of the appropriate fraction of petroleum.

[c] Where the amount of the best sample isolated was sufficient, and the sample was crystallizable, the purity has been calculated from the value of the freezing point previously reported and the present best values of the freezing point for zero impurity and cryoscopic constants from the American Petroleum Institute Research Projects 44 and 6. Where not evaluated cryoscopically, the purity has been evaluated from the physical properties or spectrographic measurements or both.

[d] The values for the amount in the crude petroleum are rounded estimates subject to revision as new data become available from the work in progress.

[e] The numbers in this column refer to the published papers of the American Petroleum Institute Research Project 6, a list of which may be obtained by writing the Petroleum Research Laboratory at the Carnegie Institute of Technology, Pittsburgh 13, Pa.

[f] Not determined.

[g] Unpublished.

[h] Determined spectrographically from measurements made in the Socony-Vacuum Laboratories, Paulsboro, N. J.

[i] Determined spectrographically from measurements made in the laboratories of the Humble Oil and Refining Co., Baytown, Tex.

[j] Determined spectrographically from measurements made in the following laboratories: Humble Oil and Refining Co., Baytown, Tex.; Socony-Vacuum Laboratories, Paulsboro, N. J.; Standard Oil Development Co., Elizabeth, N. J.; Sun Oil Co., Norwood, Pa.

[k] Purification of these samples was not carried to completion because, for purposes of identification, much purer samples were available from other sources.

[m] Identity not yet established.

[n] Tentative; identification not complete.

TABLE III.—DISTRIBUTION, BY TYPE AND BROAD FRACTION, OF THE HYDROCARBONS ISOLATED FROM ONE REPRESENTATIVE PETROLEUM.

By the API Research Project 6 as of June 30, 1953.

	Fraction				Total
	Gas	Gasoline	Kerosine	Gas-Oil	
Normal paraffins	4	6	2	5	17
Branched paraffins	1	36	...	...	37
Alkyl cyclopentanes	...	22	...	...	22
Alkyl cyclohexanes[a]	...	13	...	...	13
Alkyl benzenes	...	17	13	...	30
Alkyl cycloheptanes	...	1	...	...	1
Dicycloparaffins	...	1	2	...	3
Dinuclear aromatics	...	...	3	4	7
Aromatic-cycloparaffins	...	...	5	...	5
Total	5	96	25	9	135

[a] One of these may be an alkyl cyclopentane.

1953, by the methods referred to, and Table III gives the distribution of these 135 hydrocarbons by type and broad fraction. Table IV shows how much of each of the boiling ranges is accounted for in terms of individual compounds.

That part of the gasoline fraction of petroleum normally boiling between 40 and 132 C is substantially made up of 65 compounds. In Fig. 7, the vertical scale gives the percentage amount of this material that is constituted by any given number of the 65 hydrocarbon compounds, with the number being counted in decreasing order of occurrence, the most abundant compound being counted first, the next most abundant second, etc. As this plot shows, this gasoline material is not nearly so complex as it might have been. Five compounds constitute nearly 50 per cent of the material.

Figure 8 gives a similar plot for the entire gasoline fraction of petroleum, taken as the material normally boiling between

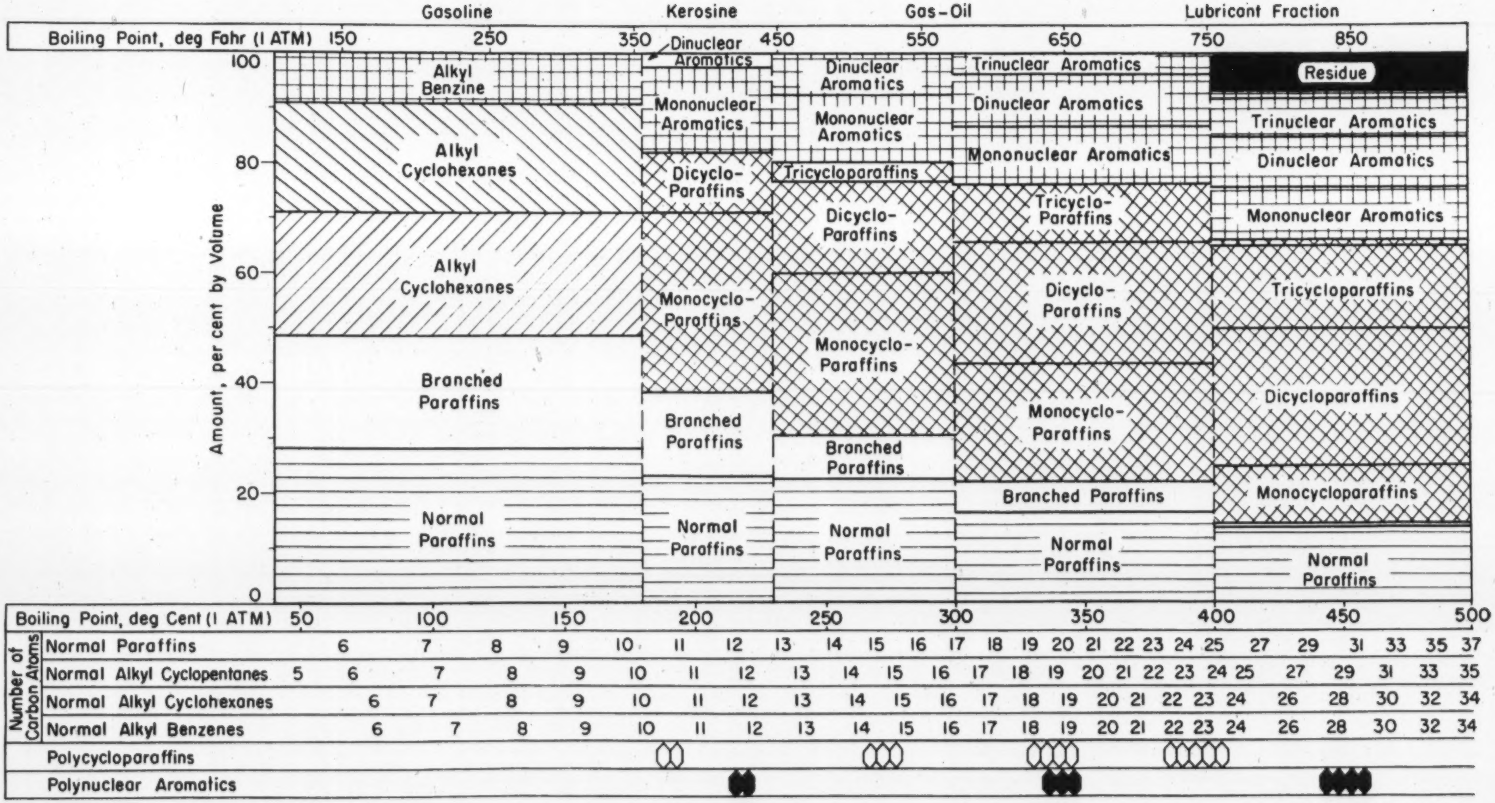

FIG. 9.—Relative Amounts of the Different Types of Hydrocarbons in the Several Broad Fractions of the Representative Petroleum of the API Research Project 6.

40 and 180 C. Here we see that although there are about 500 compounds possible in the entire gasoline fraction, the bulk of the material is made up of a relatively small number of compounds. Fifteen compounds constitute 52 per cent of the entire material. The dashed line is what we would have if Nature had made each of the 500 possible compounds occur in equal amounts in this gasoline.

TABLE IV.—SUMMARY, BY BOILING RANGE, OF THE HYDROCARBONS ISOLATED FROM ONE REPRESENTATIVE PETROLEUM.

Boiling range {deg Cent deg Fahr	<40 <104	40–180 104–356	180–230 356–446	230–300 446–572	>300 >572	Total
Compounds isolated	5	96	21	12	1	135
Estimated percentage of the fraction accounted for	>99	84	33	35	3	43

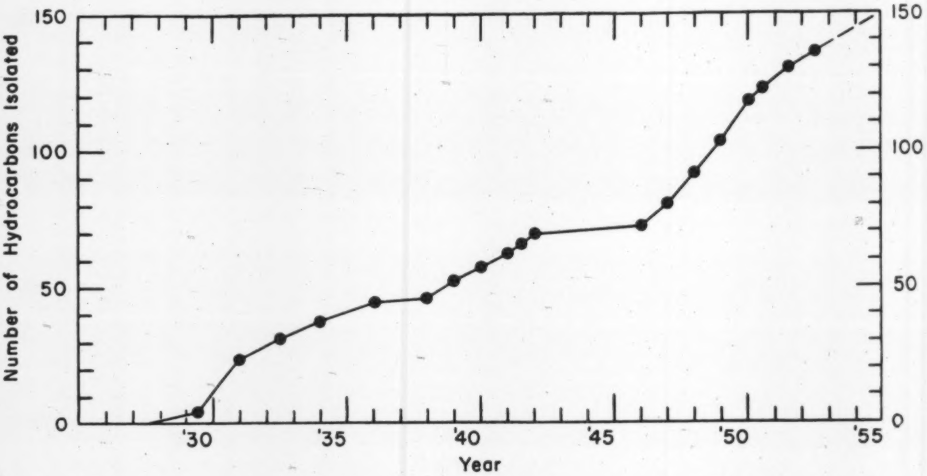

FIG. 10.—Plot of the Number of Hydrocarbons Isolated from a Large Lot of One Representative Petroleum, Plotted as a Function of the Years of Investigation.

As of June 30, 1953, the total is 135.

TABLE V.—GASOLINE FRACTIONS FROM SEVEN REPRESENTATIVE PETROLEUMS.

Source Field	Supplier	Type	Content of Sulfur, per cent by weight	Content of Aromatics, per cent by volume
Ponca, Okla.	Continental Oil Co.	Intermediate	0.017	9.8
East Texas	Standard Oil Development Co.	Intermediate	0.033	10.4
Bradford, Pa.	Quaker State Oil Refining Co.	High in paraffins	0.012	8.3
Greendale-Kawkawlin, Mich.	Pure Oil Co.	High in normal paraffins	0.014	7.2
Winkler, Tex.	Standard Oil Co. (Indiana)	High in branched paraffins	0.159	4.9
Midway, Calif.	Standard Oil Co. of California	High in alkyl cyclopentanes and alkyl cyclohexanes	0.045	8.0
Conroe, Tex.	Humble Oil and Refining Co.	High in aromatics	0.002	27.6

The composition of the various broad fractions of the Project's representative petroleum in terms of the various classes or types of hydrocarbons is summarized in Fig. 9. The work has included examination of the heavy gas-oil and lubricant fractions although no compounds have yet been separated from them.

Figure 10 shows the rate at which new hydrocarbons have been isolated from the representative petroleum of the API Research Project 6, beginning with zero in 1927 and rising to 135 as of June 30, 1953. The horizontal position of the curve near the middle corresponds in time to World War II, when the resources of the Project were largely diverted to work on military aviation fuels and related problems.

TABLE VI.—RELATIVE AMOUNTS OF THE C_6, C_7, AND C_8 AROMATIC HYDROCARBONS IN SEVEN REPRESENTATIVE PETROLEUMS.

Number of C Atoms	Compounds	Petroleum							Range of Values	Average Value
		Ponca, Okla.	East Tex.	Bradford, Pa.	Greendale-Kawkawlin, Mich.	Winkler, Tex.	Midway, Calif.	Conroe, Tex.		
6	Benzene	9	4	4	13	10	5	6	4 to 13	7
7	Toluene	30	29	31	37	20	30	38	20 to 38	31
8	Ethylbenzene *m*-Xylene *p*-Xylene *o*-Xylene	61	67	65	50	70	65	56	50 to 70	62
Total		100	100	100	100	100	100	100		100

TABLE VII.—RELATIVE AMOUNTS OF C_6 AND C_7 ALKYL CYCLOHEXANES IN SEVEN REPRESENTATIVE PETROLEUMS.

Number of C Atoms	Compounds	Petroleum							Range of Values	Average Value
		Ponca, Okla.	East Texas	Bradford, Pa.	Greendale-Kawkawlin, Mich.	Winkler, Tex.	Midway, Calif.	Conroe, Tex.		
6	Cyclohexane	30	21	24	33	31	29	30	21 to 33	28
7	Methylcyclohexane	70	79	76	67	69	71	70	67 to 79	72
Total		100	100	100	100	100	100	100		100

About 1940, the Advisory Committee for the API Research Project 6 decided that, having learned a great deal about the hydrocarbons in one representative petroleum, the Project should gain some knowledge of how petroleums from different fields varied. The Project's one representative petroleum, from Oklahoma, was intermediate in type. The gasoline fractions from six additional representative petroleums were selected to represent large production in different areas of the country and to be as different as was known at the time. The seven representative gasolines are listed together in Table V. By means of an extended but relatively short-cut procedure involving the fractionating processes of distillation and adsorption, analyses were made of the major components in these seven gasolines. From this work, the following conclusions were reached:

1. The components in the gasoline fraction of each petroleum may be placed in five main classes:

(*a*) Normal (straight chain) paraffins
(*b*) Branched paraffins
(*c*) Alkyl cyclopentanes
(*d*) Alkyl cyclohexanes
(*e*) Alkyl benzenes

2. Within each of these main classes, the individual compounds occur in proportions which are usually of the same order of magnitude for different petroleums.

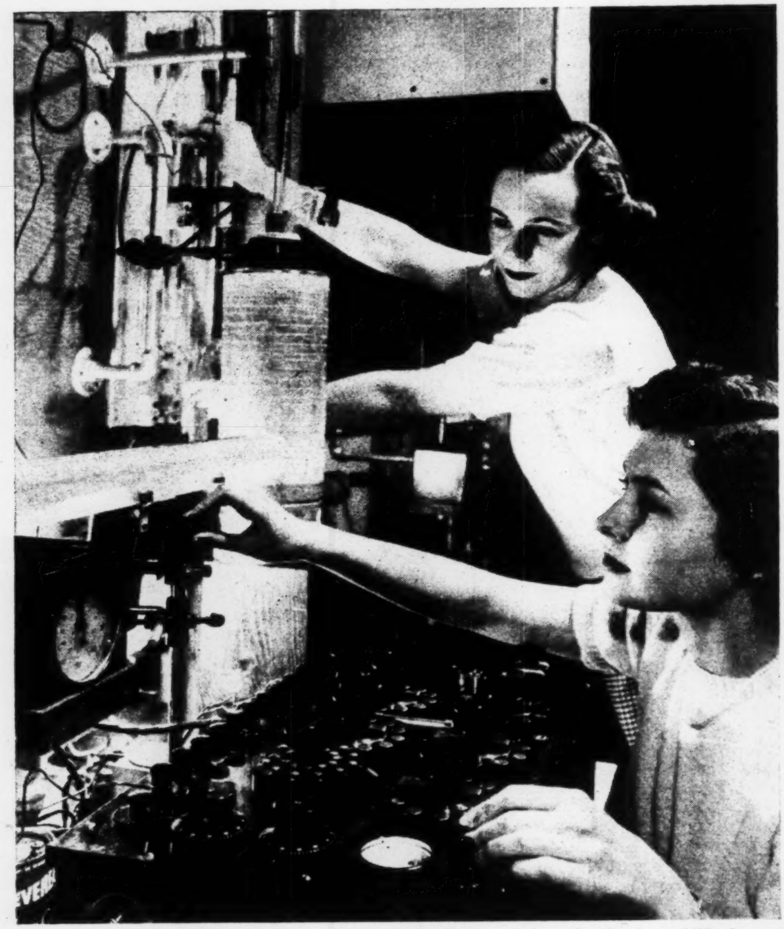

FIG. 11.—Women Chemists Serve the Industry in Determining the Purity of Hydrocarbons.

API Standard and Research hydrocarbons have their purity evaluated from accurate measurements of freezing points. With the apparatus shown here, these chemists of the API Research Project 6 at the Carnegie Institute of Technology are adjusting the freezing point apparatus and measuring temperatures to 0.001 C with platinum resistance thermometers.

The foregoing conclusions are illustrated with two examples: Table VI shows the relative amounts of benzene, toluene, and the C_8 alkyl benzenes (ethylbenzene, *o*-xylene, *m*-xylene, and *p*-xylene) in the seven representative gasolines, and Table VII shows the relative amounts of cyclohexane and methylcyclohexane. Similar relations hold for other classes of compounds and lead to important practical generalizations (**12**).

Figure 11 is a photograph showing two of our chemists determining the purity of API Standard hydrocarbons from precise measurements of freezing points. This apparatus and this procedure have formed

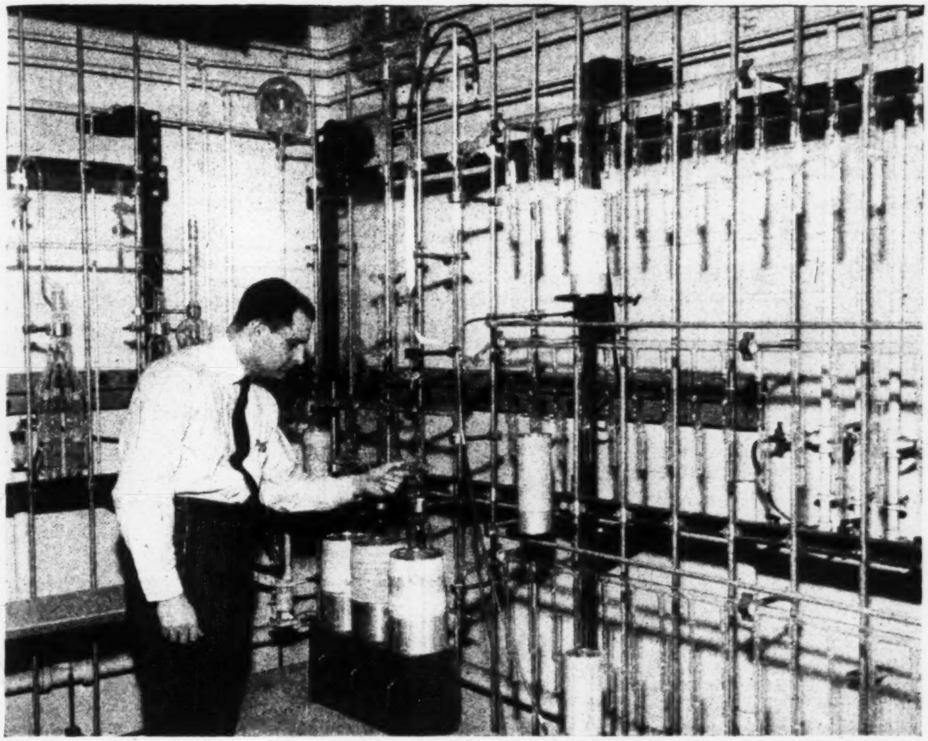

Fig. 12.—Purity Must Be Safeguarded.

Special precautions are used to preserve the purity of the API Standard and Research samples of hydrocarbons. In the apparatus shown here in the laboratories of the API Research Project 6 at the Carnegie Institute of Technology, air is removed and the highly purified hydrocarbon material is sealed "in vacuum" in small glass ampoules.

the basis of ASTM methods of test for evaluation of purity from measurements of freezing points (D 1015 and D 1016),[9, 10] developed by Section E on Determination of Purity by Freezing Point of Research Division IV on Hydrocarbon Analysis of ASTM Committee D-2 on Petroleum Products and Lubricants.

Figure 12 shows the apparatus used to seal "in vacuum" the API Standard and API Research samples of hydrocarbons. One of these sealed-up units of the API Standard hydrocarbons, a sample of 1-

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[9] Tentative Method of Test for Measurement of Freezing Points for Evaluation of Purity (D 1015 – 49 T), 1952 Book of ASTM Standards, Part 5, p. 444.

[10] Tentative Method of Test for Determination of Purity from Freezing Points (D 1016 – 52 T), 1952 Book of ASTM Standards, Part 5, p. 459.

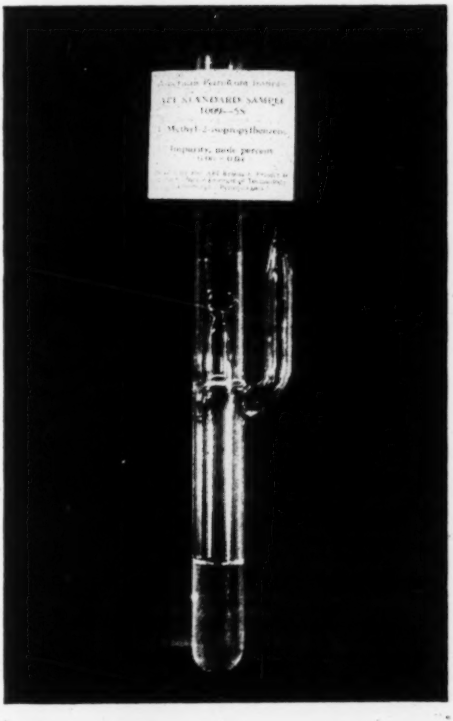

Fig. 13.—Worth Ten Times Its Weight in Gold.

The highly purified API Standard sample of 1-methyl-2-isopropylbenzene shown here is made available to the laboratories of the petroleum industry at $50 for ⅛ oz. These samples are used with spectrographic instruments to "fingerprint" hydrocarbons. From such "fingerprints," the petroleum industry determines the composition of its products and uses this information in its search for better fuels, lubricants, and other materials.

TABLE VIII.—API RESEARCH HYDROCARBONS COMPLETED AS OF JUNE 30, 1953.

Formula	Compound[a]	Purity,[b] mole per cent	Source of Starting Material[c]
	Paraffins:		
C_5H_{12}	*n*-Pentane (A)	99.85 ± 0.07	APIRP6
	n-Pentane (B)	99.95 ± 0.04	APIRP6
	2-Methylbutane (isopentane) (A)	99.89 ± 0.05	APIRP6
	2-Methylbutane (isopentane) (B)	99.95 ± 0.04	APIRP6
	2,2-Dimethylpropane (neopentane)	99.982 ± 0.012	Calif. Res. Corp.
C_6H_{14}	*n*-Hexane (A)	99.99 ± 0.01	APIRP6
	n-Hexane (B)	99.985 ± 0.010	APIRP6
	2-Methylpentane (A)	99.92 ± 0.05	APIRP6
	2-Methylpentane (B)	99.98 ± 0.01	APIRP6
	3-Methylpentane (A)	(99.90 ± 0.08)	APIRP6
	3-Methylpentane (B)	(99.90 ± 0.08)	APIRP6
	2,2-Dimethylbutane (A)	99.98 ± 0.01	APIRP6
	2,2-Dimethylbutane (B)	99.995 ± 0.005	APIRP6
	2,3-Dimethylbutane (A)	99.96 ± 0.03	Std. (Ind.); Kellogg
	2,3-Dimethylbutane (B)	99.97 ± 0.01	APIRP6
C_7H_{16}	*n*-Heptane (A)	99.94 ± 0.05	APIRP6
	n-Heptane (B)	99.99 ± 0.01	APIRP6
	n-Heptane (C)	99.99 ± 0.01	APIRP6
	2-Methylhexane	99.82 ± 0.07	Ethyl
	3-Methylhexane	(99.80 ± 0.15)	Ethyl
	3-Ethylpentane	99.94 ± 0.03	APIRP45
	2,2-Dimethylpentane	99.81 ± 0.06	Socony-Vacuum
	2,3-Dimethylpentane (A)	(99.80 ± 0.15)	General Motors
	2,3-Dimethylpentane (B)	(99.85 ± 0.10)	General Motors
	2,4-Dimethylpentane	99.88 ± 0.05	APIRP6
	3,3-Dimethylpentane	99.78 ± 0.18	APIRP45
	2,2,3-Trimethylbutane (A)	99.95 ± 0.02	General Motors
	2,2,3-Trimethylbutane (B)	99.991 ± 0.008	General Motors
C_8H_{18}	*n*-Octane	99.95 ± 0.04	APIRP6
	2-Methylheptane	99.66 ± 0.18	APIRP45
	3-Methylheptane	99.62 ± 0.23	APIRP45
	4-Methylheptane	99.89 ± 0.07	APIRP45
	3-Ethylhexane	(99.75 ± 0.20)	APIRP45
	2,2-Dimethylhexane	99.77 ± 0.11	APIRP45
	2,3-Dimethylhexane	(99.75 ± 0.20)	APIRP45
	2,4-Dimethylhexane	(99.75 ± 0.20)	Penn State
	2,5-Dimethylhexane	99.73 ± 0.09	Penn State
	3,3-Dimethylhexane	99.75 ± 0.20	APIRP45
	3,4-Dimethylhexane	(99.75 ± 0.20)	APIRP45
	2-Methyl-3-ethylpentane	99.78 ± 0.11	APIRP45
	3-Methyl-3-ethylpentane	99.93 ± 0.04	APIRP45
	2,2,3-Trimethylpentane	99.68 ± 0.20	General Motors
	2,2,4-Trimethylpentane (A)	99.94 ± 0.04	APIRP6
	2,2,4-Trimethylpentane (B)	99.95 ± 0.04	APIRP6
	2,2,4-Trimethylpentane (C)	99.97 ± 0.02	APIRP6
	2,3,3-Trimethylpentane	99.79 ± 0.08	Penn State
	2,3,4-Trimethylpentane	99.83 ± 0.06	APIRP45

[a] A letter (B), (C), or (D) following the name of a compound indicates that it is a second, third, or fourth (and usually slightly purer) sample of a given compound, the first sample of which is labeled (A).

[b] Values in parentheses are estimated.

[c] The abbreviations represent the following laboratories:
APIRP45—American Petroleum Institute Research Project 45, Ohio State Univ., Columbus, Ohio.
Penn State—Hydrocarbon Laboratory, Pennsylvania State College, State College, Pa.
APIRP42—American Petroleum Institute Research Project 42, Pennsylvania State College, State College, Pa.
APIRP6—American Petroleum Institute Research Project 6, Carnegie Institute of Technology, Pittsburgh, Pa.
Am. Cyanamid—American Cyanamid Co., Calco Chemical Div., Bound Brook, N. J.
Anglo-Iranian—Anglo-Iranian Oil Co., Ltd., Research Laboratories, Sunbury-on-Thames, England.
Atlantic—Atlantic Refining Co. Philadelphia, Pa.
Barrett—Barrett Division of the Allied Chemical and Dye Corp., New York, N. Y.
Calif. Res. Corp.—California Research Corp., Richmond, Calif.
Dow—Dow Chemical Co., Midland, Mich.
Ethyl—Ethyl Corp., Detroit, Mich.
General Motors—General Motors Corp., Detroit, Mich.
Gulf-Mellon—Gulf Oil Co. Fellowship at the Mellon Institute of Industrial Research, Pittsburgh, Pa.
Houdry—Houdry Process Corp., Marcus Hook, Pa.
Humble—Humble Oil and Refining Co., Houston, Tex.
Kellogg—M. W. Kellogg Co., New York, N. Y.
Monsanto—Monsanto Chemical Co., Dayton, Ohio.
NACA—National Advisory Committee for Aeronautics, Flight Propulsion Research Lab., Cleveland, Ohio.
NBS Auto Sec.—Automotive Section, National Bureau of Standards, Washington, D. C.
Phillips—Phillips Petroleum Co. Bartlesville, Okla.
Rubber Reserve—U. S. Office of Rubber Reserve, Washington, D. C.
Socony-Vacuum—Socony-Vacuum Laboratories, Paulsboro, N. J.
Std. Oil Dev.—Standard Oil Development Co., Elizabeth, N. J.
Std. (Ind.)—Standard Oil Co. (Indiana), Whiting, Ind.
Tide Water Assn.—Tide Water Associated Oil Co., Associated, Calif.

TABLE VIII.—*Continued.*

Formula	Compound[a]	Purity,[b] mole per cent	Source of Starting Material[c]
C_9H_{20}	*n*-Nonane	99.94 ± 0.04	APIRP6
	2,2-Dimethylheptane	99.87 ± 0.10	APIRP45
	2,2,3-Trimethylhexane	(99.75 ± 0.20)	Penn State
	2,2,4-Trimethylhexane	99.75 ± 0.20	Penn State
	2,2,5-Trimethylhexane	99.86 ± 0.04	APIRP6
	2,3,3-Trimethylhexane	99.93 ± 0.06	Penn State
	2,3,5-Trimethylhexane	(99.75 ± 0.20)	Penn State
	2,4,4-Trimethylhexane	99.73 ± 0.11	Penn State
	3,3,4-Trimethylhexane	99.87 ± 0.10	Penn State
	3,3-Diethylpentane	99.987 ± 0.011	APIRP45
	2,2,3,3-Tetramethylpentane	99.940 ± 0.020	APIRP45
	2,2,3,4-Tetramethylpentane	99.976 ± 0.014	NACA
	2,2,4,4-Tetramethylpentane	99.89 ± 0.08	NACA
	2,3,3,4-Tetramethylpentane	99.956 ± 0.037	NACA
$C_{10}H_{22}$	*n*-Decane	99.977 ± 0.018	APIRP6
	3,3,5-Trimethylheptane	(99.85 ± 0.10)	NBS Auto Sec.
$C_{11}H_{24}$	*n*-Undecane	99.97 ± 0.03	APIRP6
	2-Methyldecane	99.91 ± 0.06	APIRP45
$C_{12}H_{26}$	*n*-Dodecane	99.975 ± 0.025	APIRP6
	2,2,4,6,6-Pentamethylheptane	99.94 ± 0.04	Anglo-Iranian
$C_{13}H_{28}$	*n*-Tridecane	99.92 ± 0.06	APIRP42
$C_{14}H_{30}$	*n*-Tetradecane	99.93 ± 0.06	APIRP6
$C_{15}H_{32}$	*n*-Pentadecane	99.93 ± 0.05	Penn State
$C_{16}H_{34}$	*n*-Hexadecane	99.96 ± 0.04	APIRP6
$C_{17}H_{36}$	*n*-Heptadecane	99.91 ± 0.06	APIRP42
$C_{18}H_{38}$	*n*-Octadecane	99.90 ± 0.08	APIRP6
	Alkyl Cyclopropanes:		
C_5H_{10}	1,cis-2-Dimethylcyclopropane	99.91 ± 0.05	APIRP45
	1,trans-2-Dimethylcyclopropane	99.77 ± 0.12	APIRP45
C_6H_{12}	1,1,2-Trimethylcyclopropane	99.88 ± 0.08	APIRP45
C_7H_{14}	1,1,2,2-Tetramethylcyclopropane	99.96 ± 0.03	APIRP45
	Alkyl Cyclobutanes:		
C_6H_{12}	Ethylcyclobutane	99.92 ± 0.06	APIRP45
	Alkyl Cyclopentanes:		
C_5H_{10}	Cyclopentane (A)	99.971 ± 0.009	APIRP45
	Cyclopentane (B)	99.974 ± 0.009	Tide Water Assn.
C_6H_{12}	Methylcyclopentane (A)	99.91 ± 0.05	Houdry
	Methylcyclopentane (B)[d]	99.97 ± 0.02	Phillips
	Methylcyclopentane (C)	99.99 ± 0.01	APIRP6
	Methylcyclopentane (D)	99.99 ± 0.01	APIRP6
C_7H_{14}	Ethylcyclopentane (A)	99.89 ± 0.04	APIRP45
	Ethylcyclopentane (B)	99.92 ± 0.04	APIRP45; Penn State
	1,1-Dimethylcyclopentane	99.97 ± 0.02	Penn State
	1,cis-2-Dimethylcyclopentane	99.975 ± 0.016	Penn State
	1,trans-2-Dimethylcyclopentane	99.87 ± 0.10	Penn State
	1,cis-3-Dimethylcyclopentane	99.59 ± 0.23	APIRP45
	1,trans-3-Dimethylcyclopentane (A)	99.69 ± 0.07	APIRP45
	1,trans-3-Dimethylcyclopentane (B)	99.93 ± 0.06	APIRP45
C_8H_{16}	*n*-Propylcyclopentane	99.81 ± 0.10	APIRP45
	Isopropylcyclopentane	99.81 ± 0.07	APIRP45
	1-Methyl-1-ethylcyclopentane	99.91 ± 0.08	Penn State
	1-Methyl-cis-2-ethylcyclopentane	99.71 ± 0.24	Penn State
	1,1,2-Trimethylcyclopentane	99.988 ± 0.009	Penn State
	1,1,3-Trimethylcyclopentane	99.56 ± 0.32	APIRP45; Gulf-Mellon
	1,cis-2,cis-3-Trimethylpentane	99.93 ± 0.06	Penn State
	1,trans-2,cis-3-Trimethylcyclopentane (A)	99.92 ± 0.04	Penn State
	1,trans-2,cis-3-Trimethylcyclopentane (B)	99.90 ± 0.04	Penn State
	1,cis-2,trans-4-Trimethylcyclopentane	99.65 ± 0.23	Penn State
	1,trans-2,cis-4-Trimethylcyclopentane	99.79 ± 0.10	Penn State
C_9H_{18}	*n*-Butylcyclopentane	99.970 ± 0.025	APIRP45
	Isobutylcyclopentane	99.88 ± 0.08	APIRP45
$C_{15}H_{30}$	*n*-Decylcyclapentane	99.80 ± 0.18	APIRP45
	Alkyl Cyclohexanes:		
C_6H_{12}	Cyclohexane (A)	99.997 ± 0.002	Barrett
	Cyclohexane (B)	99.991 ± 0.006	APIRP6

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[d] The stock of this sample has been exhausted.

(Continued on pages 564 to 566.)

TABLE VIII.—*Continued.*

Formula	Compound[a]	Purity,[b] mole per cent	Source of Starting Material[c]
C_7H_{14}	Methylcyclohexane (A)	99.97 ± 0.02	Barrett; APIRP45
	Methylcyclohexane (B)	99.97 ± 0.02	APIRP6
C_8H_{16}	Ethylcyclohexane	99.90 ± 0.08	APIRP45
	1,1-Dimethylcyclohexane	99.93 ± 0.03	APIRP45
	1,cis-2-Dimethylcyclohexane	99.983 ± 0.015	APIRP45
	1,trans-2-Dimethylcyclohexane	99.92 ± 0.07	APIRP45
	1,cis-3-Dimethylcyclohexane	99.94 ± 0.05	APIRP45
	1,trans-3-Dimethylcyclohexane	99.88 ± 0.07	APIRP45
	1,cis-4-Dimethylcyclohexane	99.94 ± 0.04	Std. Oil Dev.
	1,trans-4-Dimethylcyclohexane	99.89 ± 0.08	Std. Oil Dev.
C_9H_{18}	*n*-Propylcyclohexane	99.94 ± 0.05	APIRP45
	Isopropylcyclohexane	99.90 ± 0.07	APIRP45
	1,1,3-Trimethylcyclohexane	99.85 ± 0.05	APIRP45
$C_{10}H_{20}$	*n*-Butylcyclohexane	99.958 ± 0.039	APIRP45
	Isobutylcyclohexane	99.85 ± 0.09	APIRP45
	sec-Butylcyclohexane	(99.75 ± 0.20)	APIRP45
	tert-Butylcyclohexane	99.96 ± 0.03	APIRP45
	1-Methyl-cis-4-isopropylcyclohexane	99.87 ± 0.09	APIRP45
	1-Methyl-trans-4-isopropylcyclohexane	99.97 ± 0.03	APIRP45
$C_{11}H_{22}$	2-Cyclohexyl-2-methylbutane (tert-pentylcyclohexane)	(99.85 ± 0.10)	APIRP45
$C_{16}H_{32}$	*n*-Decylcyclohexane	99.88 ± 0.11	APIRP45
	Monoolefins:		
C_5H_{10}	1-Pentene (A)	99.34 ± 0.40	Phillips
	1-Pentene (B)	99.82 ± 0.12	Phillips
	cis-2-Pentene (A)[d]	99.98 ± 0.02	APIRP45
	cis-2-Pentene (B)	99.96 ± 0.03	Phillips
	cis-2-Pentene (C)	99.99 ± 0.01	APIRP45
	trans-2-Pentene	99.93 ± 0.05	APIRP45
	2-Methyl-1-butene	99.89 ± 0.08	General Motors
	3-Methyl-1-butene (A)	99.78 ± 0.12	Houdry
	3-Methyl-1-butene (B)	99.94 ± 0.05	Phillips
	2-Methyl-2-butene	99.945 ± 0.046	General Motors
C_6H_{12}	1-Hexene	99.87 ± 0.08	APIRP45
	cis-2-Hexene	99.94 ± 0.05	APIRP45
	trans-2-Hexene	99.84 ± 0.11	APIRP45
	cis-3-Hexene	99.90 ± 0.08	APIRP45
	trans-3-Hexene	99.95 ± 0.03	APIRP45
	2-Methyl-1-pentene	99.86 ± 0.09	APIRP6
	3-Methyl-1-pentene	99.75 ± 0.20	APIRP45
	4-Methyl-1-pentene	99.85 ± 0.12	APIRP45
	2-Methyl-2-pentene	99.96 ± 0.03	APIRP45
	3-Methyl-cis-2-pentene	99.89 ± 0.08	General Motors
	3-Methyl-trans-2-pentene	99.89 ± 0.09	APIRP45
	4-Methyl-cis-2-pentene	99.93 ± 0.07	APIRP6
	4-Methyl-trans-2-pentene	99.92 ± 0.07	APIRP6
	2-Ethyl-1-butene	99.95 ± 0.04	APIRP45; APIRP6
	2,3-Dimethyl-1-butene	99.86 ± 0.13	General Motors
	3,3-Dimethyl-1-butene	99.91 ± 0.06	Penn State
	2,3-Dimethyl-2-butene	99.94 ± 0.05	General Motors
C_7H_{14}	1-Heptene	99.84 ± 0.10	APIRP45
	trans-2-Heptene	99.86 ± 0.05	APIRP45
	trans-3-Heptene	99.80 ± 0.15	APIRP45
	2-Methyl-1-hexene	99.90 ± 0.08	APIRP45
	4-Methyl-1-hexene	99.82 ± 0.16	APIRP45
	5-Methyl-1-hexene	(99.85 ± 0.10)	APIRP45
	2-Methyl-2-hexene	99.87 ± 0.11	APIRP45
	2,3-Dimethyl-1-pentene	99.85 ± 0.10	APIRP45
	4,4-Dimethyl-1-pentene	99.85 ± 0.08	APIRP45
	3-Ethyl-2-pentene	(99.85 ± 0.10)	APIRP45
	2,4-Dimethyl-2-pentene	99.88 ± 0.04	APIRP45
	4,4-Dimethyl-cis-2-pentene	99.85 ± 0.11	APIRP45
	4,4-Dimethyl-trans-2-pentene	99.91 ± 0.03	APIRP45
	2,3,3-Trimethyl-1-butene	99.95 ± 0.04	General Motors
C_8H_{16}	1-Octene	99.77 ± 0.13	APIRP6
	trans-4-Octene	99.84 ± 0.11	APIRP45
	2,3-Dimethyl-2-hexene	99.74 ± 0.09	APIRP45
	2,2-Dimethyl-cis-3-hexene	99.86 ± 0.12	APIRP45
	2,2-Dimethyl-trans-3-hexene	(99.85 ± 0.10)	APIRP45
	2,4,4-Trimethyl-1-pentene	99.95 ± 0.03	NBS Auto. Sec.
	2,4,4-Trimethyl-2-pentene	99.93 ± 0.05	NBS Auto. Sec.

TABLE VIII.—*Continued.*

Formula	Compound[a]	Purity,[b] mole per cent	Source of Starting Material[c]
C_9H_{18}	1-Nonene	99.76 ± 0.18	APIRP45
$C_{10}H_{20}$	1-Decene	99.91 ± 0.07	APIRP6
$C_{11}H_{22}$	1-Undecene	99.91 ± 0.08	APIRP45
$C_{12}H_{24}$	1-Dodecene	99.91 ± 0.07	APIRP6
$C_{13}H_{26}$	1-Tridecene	99.85 ± 0.09	APIRP42
$C_{14}H_{28}$	1-Tetradecene	99.73 ± 0.13	APIRP6
$C_{15}H_{30}$	1-Pentadecene	99.89 ± 0.05	APIRP42
$C_{16}H_{32}$	1-Hexadecene	99.93 ± 0.06	APIRP6
	Diolefins:		
C_4H_6	1,2-Butadiene	99.94 ± 0.05	Std. Oil Dev.
	1,3-Butadiene	99.92 ± 0.04	Phillips
C_5H_8	1,2-Pentadiene	99.79 ± 0.15	Penn State
	1,cis-3-Pentadiene	99.93 ± 0.04	Rubber Reserve
	1,trans-3-Pentadiene	99.97 ± 0.03	Rubber Reserve
	1,4-Pentadiene	99.94 ± 0.05	APIRP45
	2,3-Pentadiene	99.89 ± 0.07	Penn State
	2-Methyl-1,3-butadiene (isoprene)	99.96 ± 0.03	APIRP6
C_6H_{10}	1,5-Hexadiene	99.91 ± 0.08	Penn State
	2,3-Dimethyl-1,3-butadiene	99.96 ± 0.03	Penn State
	Cyclomonoolefins:		
C_5H_8	Cyclopentene	99.975 ± 0.021	Atlantic; APIRP45
C_6H_{10}	Cyclohexene	99.978 ± 0.020	APIRP6
	Acetylenes:		
C_4H_6	1-Butyne (ethylacetylene)	99.90 ± 0.07	APIRP45
	2-Butyne (dimethylacetylene)	99.959 ± 0.038	APIRP45
	Alkyl benzenes:		
C_6H_6	Benzene (A)	99.963 ± 0.020	APIRP6
	Benzene (B)	99.983 ± 0.015	APIRP6
C_7H_8	Methylbenzene (toluene) (A)	99.95 ± 0.03	Humble
	Methylbenzene (toluene) (B)	99.97 ± 0.02	Humble
C_8H_{10}	Ethylbenzene (A)	99.93 ± 0.05	Monsanto
	Ethylbenzene (B)	99.972 ± 0.020	APIRP6
	1,2-Dimethylbenzene (*o*-xylene) (A)	99.987 ± 0.012	Std. Oil Dev.
	1,2-Dimethylbenzene (*o*-xylene) (B)	99.992 ± 0.006	Std. Oil Dev.
	1,2-Dimethylbenzene (*o*-xylene) (C)	99.995 ± 0.004	Calif. Res. Corp.
	1,3-Dimethylbenzene (*m*-xylene) (A)	99.85 ± 0.05	APIRP45
	1,3-Dimethylbenzene (*m*-xylene) (B)	99.96 ± 0.03	APIRP6
	1,3-Dimethylbenzene (*m*-xylene) (C)	99.96 ± 0.03	Calif. Res. Corp.
	1,4-Dimethylbenzene (*p*-xylene) (A)	99.97 ± 0.02	APIRP45
	1,4-Dimethylbenzene (*p*-xylene) (B)	99.98 ± 0.02	Calif. Res. Corp.
C_9H_{12}	*n*-Propylbenzene (A)	99.71 ± 0.08	Dow
	n-Propylbenzene (B)	99.80 ± 0.08	APIRP6
	Isopropylbenzene (A)	99.96 ± 0.03	Monsanto
	Isopropylbenzene (B)	99.96 ± 0.03	Atlantic
	1-Methyl-2-ethylbenzene	99.76 ± 0.07	APIRP45
	1-Methyl-3-ethylbenzene[e]	99.77 ± 0.15	APIRP45
	1-Methyl-4-ethylbenzene	99.94 ± 0.03	NACA
	1,2,3-Trimethylbenzene (A)	99.80 ± 0.04	APIRP6
	1,2,3-Trimethylbenzene (B)	99.990 ± 0.009	APIRP45
	1,2,4-Trimethylbenzene (A)	99.68 ± 0.20	APIRP6
	1,2,4-Trimethylbenzene (B)	99.70 ± 0.20	NACA
	1,3,5-Trimethylbenzene	99.96 ± 0.02	APIRP45
$C_{10}H_{14}$	*n*-Butylbenzene (A)	99.91 ± 0.08	APIRP45
	n-Butylbenzene (B)	99.91 ± 0.08	NACA
	Isobutylbenzene (A)	99.86 ± 0.09	APIRP45
	Isobutylbenzene (B)	99.88 ± 0.09	NACA
	sec-Butylbenzene (A)	99.69 ± 0.06	APIRP45
	sec-Butylbenzene (B)	99.93 ± 0.06	NACA
	tert-Butylbenzene(A)	99.95 ± 0.03	APIRP45
	tert-Butylbenzene (B)	99.94 ± 0.03	NACA
	1-Methyl-2-isopropylbenzene	99.94 ± 0.04	APIRP45
	1-Methyl-3-isopropylbenzene	99.944 ± 0.038	Atlantic
	1-Methyl-4-isopropylbenzene	99.98 ± 0.02	APIRP45; NACA
	1,2-Diethylbenzene	99.97 ± 0.03	NACA
	1,3-Diethylbenzene (A)	99.92 ± 0.04	Dow
	1,3-Diethylbenzene (B)	99.95 ± 0.04	NACA; APIRP45
	1,4-Diethylbenzene	99.977 ± 0.020	NACA
	1,3-Dimethyl-5-ethylbenzene	99.92 ± 0.06	NACA
	1,2,3,5-Tetramethylbenzene	99.98 ± 0.02	APIRP45
	1,2,4,5-Tetramethylbenzene	99.92 ± 0.04	Humble

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[e] The stock of this sample has been exhausted. A new sample is in process of preparation.

(Continued on page 566.)

TABLE VIII.—*Concluded.*

Formula	Compond[a]	Purity,[b] mole per cent	Source of Starting Material[c]
$C_{11}H_{16}$	2-Phenyl-2-methylbutane (tert-pentylbenzene)	(99.85 ± 0.10)	APIRP45
	1-Methyl-3-tert-butylbenzene	99.93 ± 0.05	NACA
	1-Methyl-4-tert-butylbenzene	99.96 ± 0.03	NACA
$C_{12}H_{18}$	1,4-Diisopropylbenzene	99.98 ± 0.02	Atlantic
$C_{16}H_{26}$	*n*-Decylbenzene	99.88 ± 0.10	APIRP45
	Dicycloparaffins:		
C_9H_{16}	cis-Hexahydroindan (cis-Hydrindan)	99.95 ± 0.02	APIRP45
	trans-Hexahydroindan (trans-Hydrindan)	99.71 ± 0.11	APIRP45
$C_{10}H_{18}$	Cyclopentylcyclopentane	99.96 ± 0.03	APIRP45
	cis-Decahydronaphthalene (cis-bicyclo[4.4.0]decane)	99.93 ± 0.05	APIRP6
	trans-Decahydronaphthalene (trans-bicyclo-[4.4.0]decane)	99.97 ± 0.03	APIRP6
	Dinuclear Aromatics:		
$C_{10}H_8$	Naphthalene (A)	99.96 ± 0.03	Am. Cyanamid
	Naphthalene (B)	99.97 ± 0.03	Am. Cyanamid
$C_{11}H_{10}$	1-Methylnaphthalene	99.97 ± 0.02	APIRP6
	2-Methylnaphthalene	99.92 ± 0.06	APIRP6
	Miscellaneous:		
C_8H_{12}	4-Ethenyl-1-cyclohexene (4-vinyl-1-cyclohexene)	99.91 ± 0.07	APIRP6
C_8H_{14}	Ethenylcyclohexane (vinylcyclohexane)	99.95 ± 0.04	APIRP45
C_9H_{10}	2,3-Dihydroindene (Indan)	99.98 ± 0.02	APIRP45
$C_{10}H_{14}$	1,2,3,4-Tetrahydronaphthalene	99.93 ± 0.06	APIRP6

TABLE IX.—SUMMARY OF API RESEARCH HYDROCARBONS AS OF JUNE 30, 1953.

Class of Compounds	Number of Carbon Atoms per Molecule															Total
	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	
	Number of Different Compounds															
Normal paraffins		1	1	1	1	1	1	1	1	1	1	1	1	1	1	14
Branched paraffins		2	4	8	16	13	1	1	1							46
Alkyl cyclopropanes		2	1	1												4
Alkyl cyclobutanes			1													1
Alkyl cyclopentanes		1	1	6	10	2						1				21
Alkyl cyclohexanes			1	1	8	3	6	1					1			21
Monoolefins		6	17	14	7	1	1	1	1	1	1	1	1			52
Diolefins	2	6	2													10
Cyclomonoolefins		1	1													2
Acetylenes	2															2
Alkyl benzenes			1	1	4	8	13	3	1				1			32
Dicycloparaffins						2	3									5
Dinuclear aromatics							1	2								3
Miscellaneous					2	1	1									4
Total	4	19	30	32	48	31	27	9	4	2	2	3	4	1	1	217

methyl-2-isopropylbenzene, 99.94 mole per cent pure, is shown in Fig. 13.

A list of the 217 API Research hydrocarbons completed as of June 30, 1953, is presented in Table VIII. The API Research samples are available for loan to qualified investigators for the measurement of needed physical, thermodynamic, and spectral properties. Table IX summarizes these API Research hydrocarbons by type and number of carbon atoms. The number of API Standard hydrocarbons completed as of June 30, 1953, is 215, the same compounds as in Table VIII except for 1,1,2-trimethylcyclopropane and ethylcyclobutane. The API Standard hydrocarbons are available for purchase by the laboratories of the industry.

Figure 14 shows the apparatus used by

the API Research Project 6 for measuring densities and boiling points and vapor pressures.

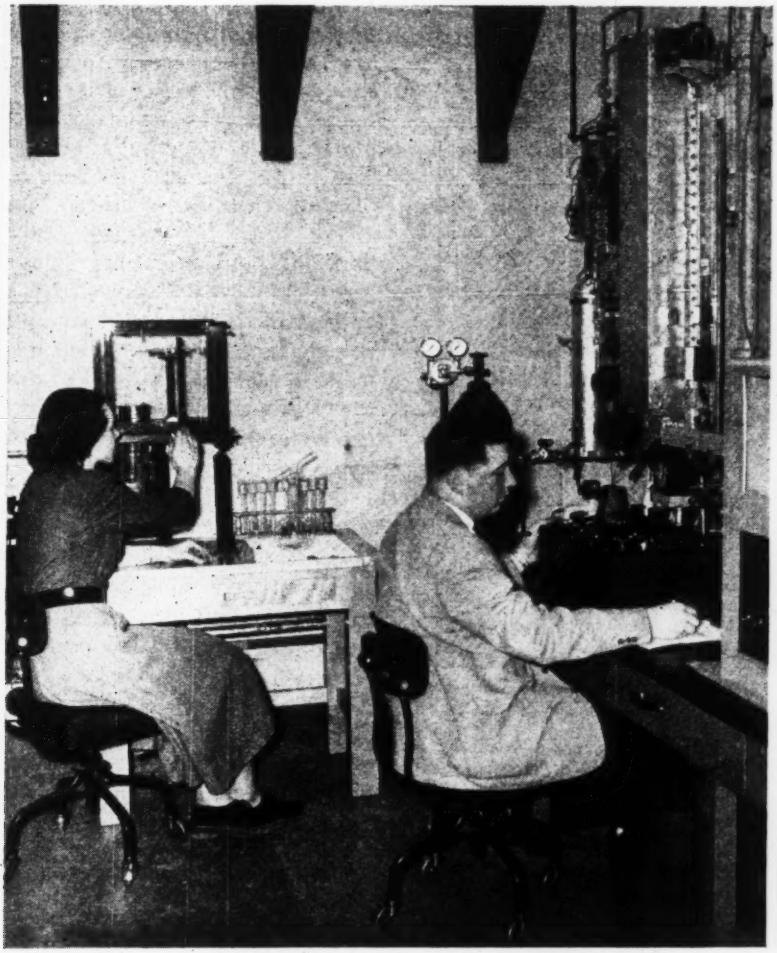

FIG. 14.—Exact Measurements Serve the Petroleum Industry.

A knowledge of the properties and behavior of hydrocarbons (the "building blocks" of petroleum) is needed by the scientists and engineers for the design of plants and the development and operation of processes. The two apparatuses shown here in the laboratories of the API Research Project 6 at the Carnegie Institute of Technology are for making accurate measurements of density (at the left) and boiling points and vapor pressures (at the right).

API RESEARCH PROJECT 44

The investigations of the API Research Project 44 are aimed to make available to the laboratories of the petroleum industry, and to the technical world in general, all of the data on hydrocarbons and related compounds in the form of tables of selected values of physical and thermodynamic properties, and catalogs of infrared, ultraviolet, Raman, and mass spectral data.

The entire available data are examined, appraised, and correlated. Original calculations are made, as necessary. "Best" values are selected. The data are put into useful form and distributed nationally and internationally to the free world. Descriptions of the original methods and calculations are published in scientific journals. And, finally, the tables of physical and thermodynamic properties are maintained up to date by revision at appropriate intervals.

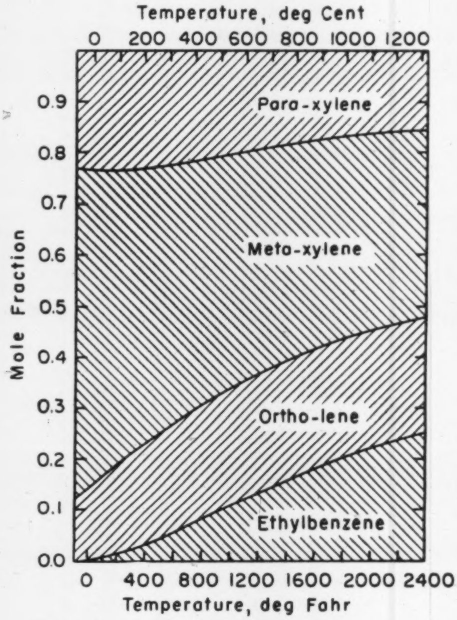

FIG. 15.—Relative Amounts of the Four C_8 Aromatic Hydrocarbons at Thermodynamic Equilibrium in the Gas Phase at Different Temperatures.

The relative amount of any one component is measured by the vertical width of its band at the given temperature.

One of the complications arising in the work on hydrocarbons is the existence of so many isomers. For example, there are 35 different nonanes, C_9H_{20}, and 75 different decanes, $C_{10}H_{22}$. Some of the isomers have widely differing properties.

By means of suitable correlations of properties with molecular structure, it is possible to obtain reliable values of properties for compounds that have never yet been prepared in any laboratory. Appropriate correlations with temperature (or presssure) serve to yield values for temperatures (or pressures) not before measured.

Figure 15 shows a chart derived from the thermodynamic data compiled by the API Research Project 44. This chart shows the relative amounts of each of the four C_8 alkyl benzenes at thermodynamic equilibrium in the gas phase at different temperatures. The compounds included in the chart are ethylbenzene (a component for synthetic rubber), *p*-xylene (a component for synthetic fibers), *o*-xylene (used for making phthalic anhydride), and *m*-xylene. The vertical width of a band gives the relative amount of that isomer at equilibrium at the given temperature. The predictions made by this chart as to the amounts of the C_8 aromatics produced in our modern catalytic cracking processes have been amply verified by experience.

Consider, briefly, the thermodynamic equilibrium between *m*-xylene, with the two methyl groups on the benzene ring in the 1 and 3 positions, and *p*-xylene, with the two methyl groups on the benzene ring in the 1 and 4 positions:

$$C_8H_{10}\ (\text{gas, } m\text{-xylene}) = C_8H_{10}\ (\text{gas, } p\text{-xylene})$$

The equilibrium constant, K, for this reaction is the ratio of the concentration or partial pressure of *p*-xylene to that of *m*-xylene:

$$K = \frac{P_{p\text{-xylene}}}{P_{m\text{-xylene}}}$$

But from chemical thermodynamics, we know that

$$\Delta F^\circ = -RT\ln K$$

and

$$\Delta F^\circ = \Delta H^\circ - T\Delta S^\circ$$

where, for any given process or reaction, ΔF° is the standard change in free energy, ΔH° is the standard change in heat content or enthalpy, and ΔS° is the standard change in entropy (**11**). Combination of the last two equations, with elimination of ΔF°, yields

$$\ln K = \frac{\Delta H^\circ}{RT} - \Delta S^\circ$$

or, writing K explicitly,

$$K = e^{-\Delta H^\circ/RT}\, e^{\Delta S^\circ/R}$$

TABLE X.—SUMMARY OF THE INDIVIDUAL COMPOUNDS IN THE TABLES OF PHYSICAL AND THERMODYNAMIC PROPERTIES OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

Group Number	Class of Compounds, from Title	Number of Individual Compounds
00	O, H, N, C	4
0	O_2, H_2, OH, H_2O, N_2, NO, C, CO, CO_2	9
1	Paraffins, C_1 to C_5	8
2	Paraffins, C_6 and C_7	14
3	Paraffins, C_8	18
4	Paraffins, C_9	35
5	Alkyl benzenes, C_6 to C_9	14
6	Alkyl cyclopentanes, C_5 to C_7	8
7	Alkyl cyclohexanes, C_6 to C_8	10
8	Monoolefins, C_2 to C_6	29
9	Monoolefins, C_7	36
10	Monoolefins, C_8	92
11	Diolefins, C_3 to C_6	32
12	Acetylenes, C_2 to C_5	7
13	Styrenes, C_8 and C_9	7
14	Alkyl benzenes, C_{10}	22
15	Alkyl cyclopentanes, C_8	15
17	Paraffins, C_{10}	75
18	Alkyl cyclopentenes, C_5 to C_7	17
19	Alkyl cyclohexenes, C_6 to C_8	22
20	Normal paraffins, C_1 to C_{40}	40
21	Normal alkyl benzenes, C_6 to C_{42}	37
22	Normal alkyl cyclopentanes, C_5 to C_{41}	37
23	Normal alkyl cyclohexanes, C_6 to C_{42}	37
24	Normal monoolefins (1-Alkenes), C_2 to C_{40}	39
25	Normal acetylenes (1-Alkynes), C_2 to C_{40}	39
26	Alkyl benzenes, C_{11}	51
27	Naphthalenes, C_{10} to C_{12}	15
28	Tetrahydronaphthalenes, C_{10} to C_{12}	31
29	Decahydronaphthalenes, C_{10} to C_{12}	68
30	1-Normal alkyl naphthalenes, C_{10} to C_{22}	13
31	2-Normal alkyl naphthalenes, C_{10} to C_{22}	13
101	1-*n*-Alkanethiols, C_1 to C_{20}	20
102	2-Alkanethiols, C_3 to C_{20}	18
103	Alkanethiols, C_1 to C_5	16
104	Alkyl benzenethiols, C_6 to C_8	13
105	2-*n*-Thiaalkanes, C_2 to C_{20}	19
107	Thiaalkanes, C_2 to C_5	11
108	Thiaalkanes, C_6	14
109	Alkyl (1-thiaalkyl) benzenes, C_7 and C_8	5
110	Alkyl (1-thiaalkyl) benzenes, C_9	14
111	Alkyl thiacyclopropanes, C_2 to C_4	6
112	Alkyl thiacyclopentanes, C_4 to C_6	15
113	Alkyl thiacyclohexanes, C_5 to C_7	22
114	Alkyl thiophenes, C_4 to C_6	9
115	Alkyl thiophenes, C_7	12
Total		1088
Number of duplications		58
Total number of different individual compounds		1030

The equilibrium constant thus becomes expressed as the product of two terms, one of which calls for minimum heat content or approximately energy, and the other maximum entropy. Minimum energy arises in a molecule when its atoms are put together in such a way that the atoms are bound together most strongly, leading to maximum security. Maximum entropy arises for a molecule when its atoms are put together in a way such that the molecule has the maximum

TABLE XI.—SUMMARY OF INDIVIDUAL NUMERICAL ENTRIES IN THE LOOSE-LEAF DATA SHEETS ON PHYSICAL AND THERMODYNAMIC PROPERTIES OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

Letter Designation of Tables	Properties for Which Values Are Tabulated	Number of Individual Numerical Entries for All Compounds
α	Values of constants	77
β	Conversion factors	421
γ	Useful equations with numerical constants	11
δ	Molecular weights	6 318
a	Boiling point, dt/dp, refractive index, density, and freezing point	6 009
a–E	Boiling point, dt/dp, refractive index, density, and freezing point	10 485
b	Molecular volume, molecular refraction, specific refraction, refractivity intercept, and specific dispersion	8 648
c	Viscosity (absolute)	3 070
c–E	Kinematic viscosity	2 752
c–K	Kinematic viscosity	3 073
d	Density	1 276
d–E	Density	2 226
e	Surface tension	824
i	Critical temperature, pressure, density, volume, and compressibility factor	1 000
k	Vapor pressures and boiling points	7 486
k–E	Vapor pressures and boiling points	11 956
m	Heat and entropy of vaporization	1 046
n	Heat of combustion	2 268
p	Energy of formation, entropy, and free energy of vaporization	990
q	Standard heat, entropy, and free energy of vaporization	202
r	Heat energy function	3 985
s	Free energy function	4 001
t	Entropy	3 981
u	Heat content	3 985
u–E	Heat content	6 830
u–G	Heat content	3 651
v	Heat capacity	3 985
v–E	Heat capacity	6 824
v–G	Heat capacity	3 648
w	Heat of formation	3 797
x	Free energy of formation	3 811
y	Logarithm of equilibrium constant of formation	3 807
z	Heat and entropy of fusion, freezing points, and cryoscopic constants	868
		123, 311

TABLE XII.—SUMMARY OF THE STATUS OF THE CATALOG OF INFRARED SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

NUMBER AND KINDS OF SHEETS IN THE CATALOG

	Number of Sheets
Total number of contributions of spectral data	1447
Descriptions of spectral equipment	34
Title and index	95
Explanatory text	1
Total	1577

NUMBER AND KINDS OF COMPOUNDS IN THE CATALOG

	Number of Different Compounds
Hydrocarbon compounds, C_1 to C_{20}	374
Hydrocarbon compounds, over C_{20}	70
Total hydrocarbon compounds	444
Compounds containing no C	4
Compounds of O, C, etc	61
Compounds of F, Cl, Br, I, C, etc	98
Compounds of N, C, etc	22
Compounds of S, C, etc	108
Miscellaneous	8
Total nonhydrocarbon compounds	301
Total compounds	745

TABLE XIV.—SUMMARY OF THE STATUS OF THE CATALOG OF RAMAN SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

NUMBER AND KINDS OF SHEETS IN THE CATALOG

	Number of Sheets
Total number of contributions of spectral data	214
Title and index	22
Descriptions of apparatus and experimental procedure	4
Total	240

NUMBER AND KINDS OF COMPOUNDS IN THE CATALOG

	Number of Different Compounds
Hydrocarbon compounds, C_1 to C_{20}	87
Hydrocarbon compounds, over C_{20}	0
Total hydrocarbon compounds	87
Compounds containing no C	2
Compounds of O, C, etc	55
Compounds of F, Cl, Br, I, etc	52
Compounds of N, C, etc	1
Compounds of S, C, etc	14
Total nonhydrocarbon compounds	124
Total compounds	211

TABLE XIII.—SUMMARY OF THE STATUS OF THE CATALOG OF ULTRAVIOLET SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

NUMBER AND KINDS OF SHEETS IN THE CATALOG

	Number of Sheets
Total number of contributions of spectral data	545
Title and index	42
Explanatory text	1
Total	588

NUMBER AND KINDS OF COMPOUNDS IN THE CATALOG

	Number of Different Compounds
Hydrocarbon compounds, C_1 to C_{20}	150
Hydrocarbon compounds, over C_{20}	25
Total hydrocarbon compounds	175
Compounds containing no C	0
Compounds of O, C, etc	61
Compounds of F, Cl, Br, I, C, etc	20
Compounds of N, C, etc	35
Compounds of S, C, etc	73
Miscellaneous	2
Total nonhydrocarbon compounds	191
Total compounds	366

TABLE XV.—SUMMARY OF THE STATUS OF THE CATALOG OF MASS SPECTRAL DATA OF THE API RESEARCH PROJECT 44 AS OF JUNE 30, 1953.

NUMBER AND KINDS OF SHEETS IN THE CATALOG

	Number of Sheets
Total number of contributions of spectral data	940
Title and index	63
Descriptions of apparatus and experimental procedure	15
Explanatory text	1
Total	1019

NUMBER AND KINDS OF COMPOUNDS IN THE CATALOG

	Number of Different Compounds
Hydrocarbon compounds, C_1 to C_{20}	376
Hydrocarbon compounds, over C_{20}	63
Total hydrocarbon compounds	439
Compounds containing no C	11
Compounds of O, C, etc	128
Compounds of F, Cl, Br, I, C, etc	68
Compounds of N, C, etc	40
Compounds of S, C, etc	63
Miscellaneous	5
Total nonhydrocarbon compounds	315
Total compounds	754

TABLE XVI.—DISTRIBUTION OF LOOSE-LEAF DATA SHEETS OF THE API RESEARCH PROJECT 44 TO GRATIS RECIPIENTS IN THE UNITED STATES AS OF JULY 1, 1953.

Kinds of Institutions	Number of Institutions	Number of Individual Recipients	Number of Sets					
			Physical and Thermodynamic Properties	Infrared Spectral Data	Ultraviolet Spectral Data	Raman Spectral Data	Mass Spectral Data	Total
Universities	140	216	185	160	150	135	124	754
Independent libraries	5	5	4	5	5	4	4	22
Non-profit research	15	15	13	13	12	12	10	60
Government	65	71	56	55	50	38	39	238
Total	225	307	258	233	217	189	177	1074

TABLE XVII.—DISTRIBUTION OF LOOSE-LEAF DATA SHEETS OF THE API RESEARCH PROJECT 44 TO GRATIS RECIPIENTS ABROAD, INCLUDING UNIVERSITIES, NON-PROFIT RESEARCH INSTITUTIONS, AND GOVERNMENT INSTITUTIONS, AS OF JULY 1, 1953.

Country	Number of Institutions	Number of Individual Recipients	Number of Sets					
			Physical and Thermodynamic Properties	Infrared Spectral Data	Ultraviolet Spectral Data	Raman Spectral Data	Mass Spectral Data	Total
Anglo-Egyptian Sudan	1	1	1	1	1	1	1	5
Argentina	2	2	2	2	2	2	2	10
Australia	6	11	8	8	8	5	5	34
Belgium	3	6	7	6	6	4	4	27
Brazil	2	2	1	1	1	1	1	5
Canada	8	11	7	8	7	6	10	38
Denmark	2	2	2	2	2	2	1	9
France	8	8	7	7	6	4	5	29
Germany (West)	10	11	9	7	7	7	7	37
Great Britain	31	45	34	35	33	30	28	160
India	10	10	9	8	7	7	7	38
Ireland	2	2	2	2	2	2	2	10
Israel	1	1	1	1	1	1	1	5
Italy	4	4	4	4	4	4	4	20
Japan	5	5	3	3	3	3	4	16
Lebanon	1	1	1	1	1			3
Netherlands	7	8	7	8	8	7	6	36
New Zealand	3	3	2	3	3	3	1	12
Norway	2	2	2	2	1	1	1	7
Philippine Islands	1	1	1					1
South Africa	3	3	2	2	2	2	3	11
Spain	5	5	4	4	3	4	3	18
Sweden	6	6	6	4	5	3	3	21
Switzerland	4	5	5	4	4	4	4	21
Yugoslavia	1	1		1				11
Total	128	156	127	124	117	103	103	574

number of states of existence, leading to maximum freedom. We see then that in every chemical process the state of equilibrium is a compromise between these two more or less opposing tendencies, toward maximum security on the one hand and maximum freedom on the other.

The 1030 hydrocarbons and related compounds for which values of physical and thermodynamic properties are given in the tables of the API Research Project 44 as of June 30, 1953, are listed in Table X by groups.

Table XI summarizes the properties for which values are given in the tables of the API Research Project 44 as of June 30, 1953, when the tables contained 123,311 individual numerical entries.

Values for P-V-T and related properties are being investigated and will appear in the tables in the near future.

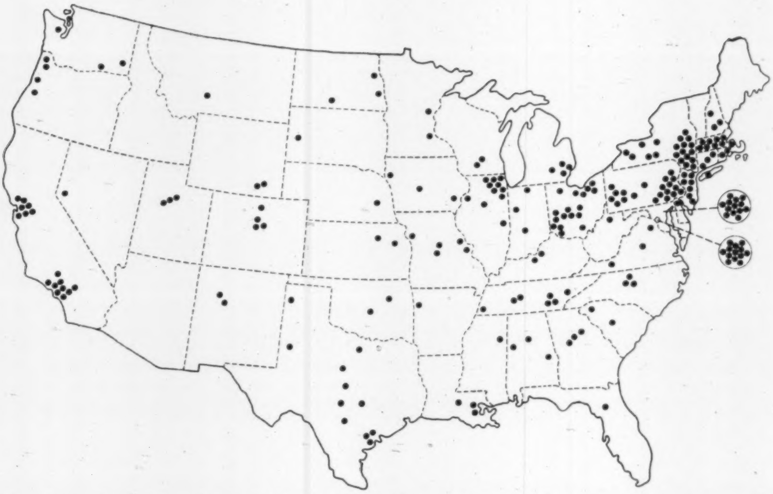

FIG. 16.—Location of Institutions in the United States Receiving Loose-Leaf Data Sheets of the API Research Project 44 on a Gratis Basis.

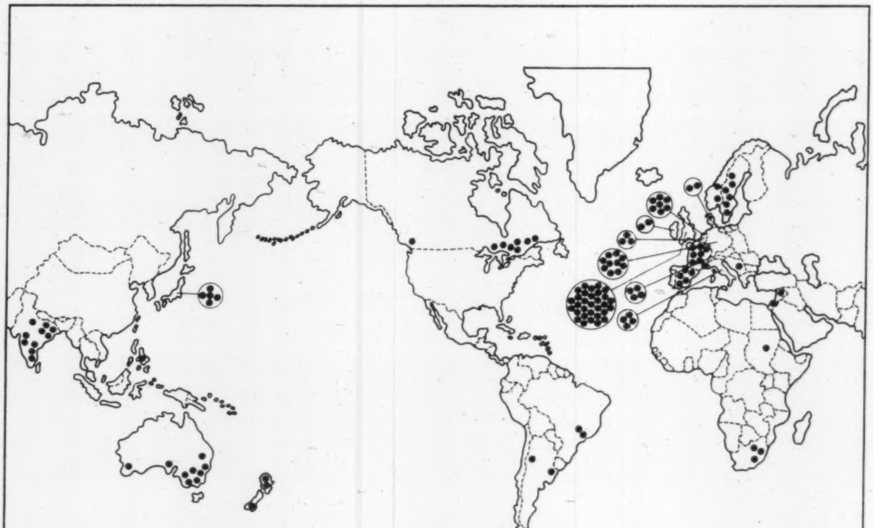

FIG. 17.—Location of Institutions in the Free World Receiving Loose Leaf Data Sheets of the API Research Project 44 on a Gratis Basis.

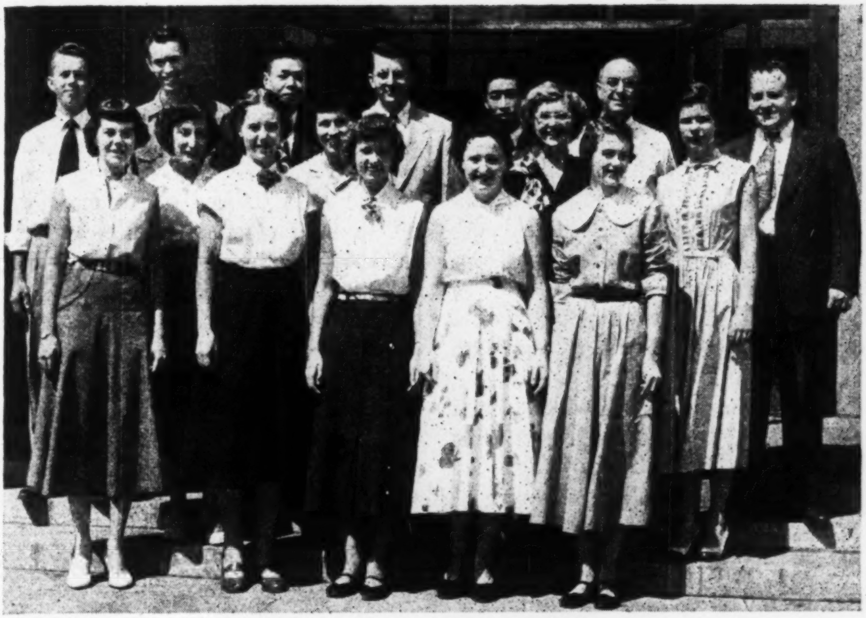

FIG. 18.—Staff of the API Research Project 44, on August 26, 1952.

Back row: Rudolph J. Pallan, Norman A. Leister, Shui-Shen Hu, Harry J. Ries, Kun Li, Frederick D. Rossini Lawrence N. Canjar

Middle row: Rita M. Braun, Janet Mitchell, Evelyn V. Gianni, Shirlee J. Ploeger

Front row: Louise A. Parcella, Phyllis A. Cowie, Marcella M. Prusak, Sara M. Spoltore, Mary C. Hinman

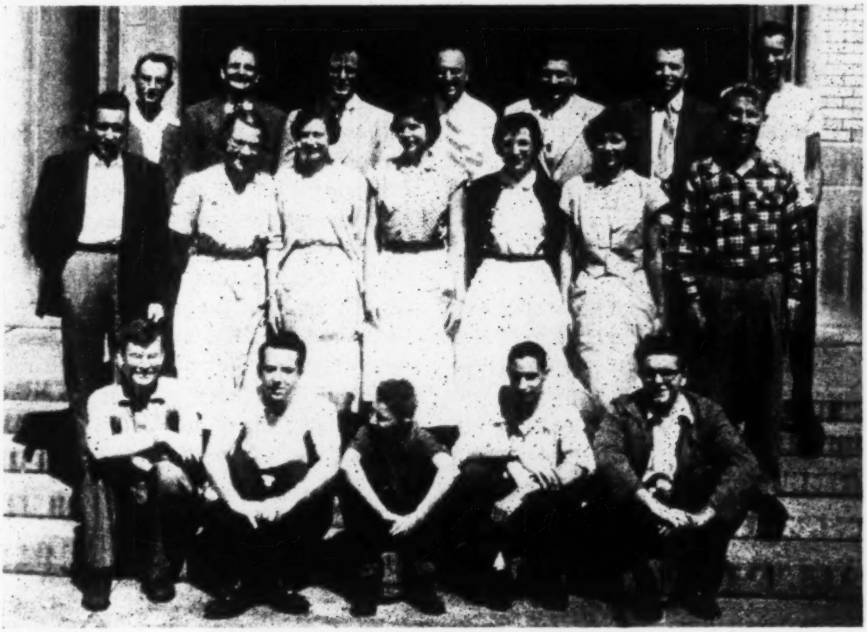

FIG. 19.—Staff of the API Research Project 6, on August 1, 1952.

Back row: Frank V. Pleasant, Anton J. Streiff, Beveridge J. Mair, Frederick D. Rossini, David L. Camin, Ned C. Krouskop, and Norman A. Leister

Middle row: Speros D. Mandamadiotis, Lillian C. Janicik, Mary C. Hinman, Shirlee J. Ploeger, M. Elizabeth Janes, Marcella M. Prushak, and William J. Marculaitis

Front row: Wilm E. Donath, Albert Berkovitz, Frederick A. Rossini, David Mikszan, and Clyde A. Weber

Tables XII to XV give summaries of the status of the various catalogs of spectral data of the API Research Project 44 as of June 30, 1953. Contributions to these catalogs have been made by laboratories of the petroleum industry, government, and schools. Forty-three different laboratories contributed to the infrared, 32 to the ultraviolet, 9 to the Raman, and 18 to the mass spectral data catalogs.

The distribution of loose-leaf data sheets of the API Research Project 44 to gratis recipients in the United States and abroad, as of July 1, 1953, is shown in Tables XVI and XVII, respectively.

Figure 16 shows graphically the location of the gratis recipients of the loose-leaf data sheets of the API Research Project 44 in the continental United States, including universities, libraries, non-profit research institutions, and government laboratories, as of July 1, 1953.

Figure 17 shows graphically the location of the gratis recipients of the loose-leaf data sheets of the API Research Project 44 in the world outside the continental United States, including universities, non-profit research institutions, and government laboratories, as of July 1, 1953.

CONCLUSION

More than 400 man-years of work have gone into the API Research Projects 6 and 44. Many research investigators and assistants have come into the work and graduated from it. The accomplishments are due in large part to the coordinated team work of the staff. While I cannot here give adequate acknowledgment to all those who have contributed to the work, I am glad to present the recent staff. Figures 18 and 19 are photographs of the staff of the API Research Projects 44 and 6, respectively, as of last fall. Those who were absent when these pictures were taken, or who have since joined the staff, include the following: Lawrence H. Schultz, Aura Raspaldo Hulme, Mary Ellen Cristina, John B. Greenshields, Monty J. Montjar, Arthur Pignocco, Shirley McIlree Butt, Kathryn Chieffalo, and James R. Ward.

More than 400 man-years of work have gone into the API Research Projects 6 and 44. Many research investigators and assistants have come into the work and have been graduated from it, and the accomplishments are due in large part to the coordinated teamwork of this staff.

I would like to close my talk with several simple statements concerning research:

Scientific research is one of the most important endeavors in our present-day civilization. By its very nature, research cannot be planned in advance with the precision of a process for mass production. The greatest fascination of research lies in the work on the frontiers of science, where investigators are probing into what is for man the unknown. Here are discovered the facts and laws of Nature, some expected and others wholly unexpected, as laid down in such beautifully simple patterns by our God, the Creator of all.

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GILLETT MEMORIAL LECTURE

THE GILLETT MEMORIAL LECTURE is sponsored jointly by the American Society for Testing Materials and Battelle Memorial Institute in commemoration of Horace W. Gillett, the subjects of the Lectures to pertain to the development, testing, evaluation, and application of metals.

MICROMETALLURGY—THE METALLURGY OF MINUTE ADDITIONS[1]

SECOND GILLETT MEMORIAL LECTURE

BY JEROME STRAUSS[2]

The preparation and presentation of this Second Gillett Memorial Lecture has deep connotations. Not only are distinction and honor represented but significantly a great measure of responsibility. I sense this responsibility as much because the invitation comes from two great institutions as from my deep respect for Dr. Gillett both as a man and as a technologist. The fact that I have had personal contact with certain of his accomplishments adds interest and enjoyment to the whole.

N. L. Mochel covered so ably and so thoroughly in the First Lecture the qualities of the man, the nature and volume of his contributions to metallurgical science and metallurgical practice that it may seem improper or superfluous to add to this story. Perhaps, however, I shall ultimately be forgiven for a few additions in view of my association with them and moreover the connection between one of them and the subject matter of this lecture.

From the personal side, a large proportion of this audience knew of Dr. Gillett's interest in dogs and in *his* dogs in particular, but it should not go unrecorded that his Christmas cards always carried a canine flavor, always the Gillett dogs, often Gillett photographs, and almost always Gillett poetry. No doubt some of you, heartened even as I was by these bits of originality, will remember my favorite—just a single photograph showing Gil with a soft hat drawn down over his face, the dog with paws on his shoulder nuzzling under the hat, and the caption "I am telling my master (he's the guy under the hat) to wish you a Merry Xmas and a Happy New Year for both of us" signed "Flash" Gillett. How characteristic also of Gil's writing, not only in personal messages but even when dealing with a "highbrow" subject.

It was my great good fortune to have known the man whose memory we honor today since the very early twenties. A small group was at that time organized in Washington as the Metals Specifications Board, later to become the nucleus of the Federal Specifications Board. Dr. Gillett came from the U. S. Bureau of Mines Station at Ithaca to attend its meetings. His comments, cheerfully given yet often blunt and incisive, at times left both chairman and secretary somewhat aghast. Just a few years later he became a Washington resident, and it was not long thereafter that I learned to respect his ability to scan quickly a mass of scattered or only partly related data and, like his dogs, scent the most profitable direction of further attack upon the problem in hand. Later, this ability developed into a strong liking, perhaps even a passion, for a type of metallurgical research to which I shall shortly refer.

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[1] Presented at the Fifty-sixth Annual Meeting of the Society June 30, 1953, Atlantic City, N. J.

[2] Vice President, Vanadium Corporation of America, New York, N. Y.

This Washington activity lasted only five years, but for some of us it meant contact for only four, as between 1928 and 1929 four close associates in the metallurgical field took up other activities—in reverse chronological order Dr. Gillett, the late P. E. McKinney, H. J. French, and I. How well I remember a meeting in 1929 of the Washington Chapter of the then American Society for Steel Treating, shortly following this exodus and called the "Four Horsemen Meetting," at which for obvious reasons steel-making alloys were discussed and Dr. Gillett, still enthusiastic over the publication of the manuscript (with the late E. L. Mack) of "Molybdenum, Cerium, and Related Alloy Steels," **(1)**[3] held forth for molybdenum. Despite my new association, and hence discussion of another element, I could not restrain some sympathetic response in view of participation in some of the tests recorded in that volume.

It was in the same year that, in my office, the late F. M. Turner, Jr., of the then Chemical Catalog Co. advanced the idea of a new periodical on metallurgy to the late Dr. B. D. Saklatwalla and me. After discussion of coverage, came the query of who could best undertake editorial direction. The answer was prompt and unanimous. There was only the question as to whether, with his new responsibilities, Dr. Gillett could and would. You all know that he did.

In these few historical notes, mention has been made of Dr. Gillett's invariably quick grasp of the relation between numerous data and concepts dealing with either specific problems or broad subjects and of his keen interest in delving into source material, critically evaluating it, and setting forth in written form the significant aspects of some metallurgical facet that at the moment had great importance or special and specific interest. The subjects he chose, appearing in *Metals and Alloys* as "Critical Abstracts" attracted wide attention, for they touched upon problems common to large segments of the profession; likewise did his treatment of these subjects bring information and assistance, recognized and acknowledged by great numbers of his audience.

It was in 1949, following the realization that published data on the corrosion resistance of very-low-carbon austenitic corrosion-resistant steels presented sharp disagreements and had led to widely varying opinions as to their probable performance, that Committee A-10 of this Society conducted a symposium, arranged by F. L. LaQue, in an effort to clarify these disagreements and point to accepted knowledge and the features on which precise data were lacking. It was my privilege to request Dr. Gillett to prepare the opening paper for that meeting and in it to present critically the state of published material up to that moment. He responded immediately and in the affirmative, but, greatly to be regretted, he was to prepare no more constructively suggestive digests of this kind.

It is my intent today to review and to comment upon the growing number of major effects upon metals and alloys resulting from or associated with minute changes in composition, both additive and the reverse, many of them dealing with quantities of a minor element ever so much smaller than the content of carbon in the steels forming the subject of the controversy in which Dr. Gillett's last paper served so useful a purpose.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 594.

Not that many of you are not well acquainted with some of them, but it is my hope that bringing a large number of them to your attention simultaneously and treating at least some in a critical fashion, may stimulate others to extend further our knowledge of these most interesting phenomena. The scope of this abstract—and I hope that it may approach in quality and value the "Critical Abstracts" that we so greatly miss—can range across the full field of metallurgical practice, for such is the scope of this Society's interests and was the scope of Dr. Gillett's interests and of Battelle Memorial Institute which he launched on its eminent path.

This is by no means the first occasion on which the influence on metals and alloys of small additions of other metals or other elements has been reviewed, but I believe it to be the first devoted entirely to exceedingly small percentages over a very broad range of metallurgical experimentation and practice. The subject is brought to the fore by two striking instances—the behavior of certain semiconductors that have led to the development of the transistor, now effecting major changes in the communications industry, and the development of steels treated with boron-containing alloys that have taken and will retain their place in the steel industry and which at the moment, due only in part to shortages of some of the conventional alloying elements, account for 10 per cent of the total alloy steel production of the United States. But there are many other examples, more or less related, possessing more or less industrial importance, some old, some new.

Of course, everyone whose activities either touch upon or are deeply immersed in the metallurgical industry can bring forth, without a moment of hesitation, illustrations of the damaging effects of very minor amounts of impurities on the characteristics of metals and alloys. The list of undesirables would include gases, non-metals, and also metals. I propose not to discuss this phase of a very broad subject although it is difficult to avoid it in view of the fact that many enhancements of properties may (when the mechanisms are clear) merely mean that these "impurities," if such they be, have been removed, or rendered innoccuous, or converted to useful forms by the addition of other substances in amounts of the same order of magnitude. Essentially I shall treat of beneficial additions, even though, with in many cases the mechanisms of their behavior in doubt at the moment, they are regarded by some in the nature of correctives for the detrimental behavior of other elements. Use obviously dictates required properties and one cannot overlook that qualities damaging to one application are greatly desired for another. When the purposes are different, an intolerable curse at one level of content may convert to a rare blessing at another; well recognized examples are sulfur and phosphorus in some steels (2,3,4,5).

Reverting to the older examples of very small additions, one is reminded of the effects of advances in the preparation of metals of high purity.

Copper is among the metals once regarded as readily obtainable commercially in very "pure" form. When in 1913 the International Annealed Copper Standard was established, the commercially pure copper that it described, which was everywhere available, carried slightly in excess of 99.9 per cent copper. Metal of higher purity (close to but not quite reaching 99.99 per cent) had been prepared to yield conductivity values somewhat over 101 per cent of this standard. Today's commercial product, if, oxygen-free, approaches this purity or, with oxygen present, is not far below.

From the beginning there were considerable data on the effects of the addition of numerous elements upon this prime property—conductivity. One result was the purposeful incorporation of silver to a total of at least 0.05 per cent to increase the softening temperature of the metal without adversely affecting conductivity, thus meeting the demand for resistance to deformation at moderately elevated temperature in a number of applications. Only in the last 10 or 15 years has this early knowledge undergone revision and the precise new information, springing from experimental production of metal of "super-purity" and the application of new methods of measurement and control, opened a vista of new means to achieve an old objective. With this metal of 99.999+ per cent purity and the very high conductivity of 102.3 per cent, came the opportunity to explore far smaller amounts of addition agents than were previously determinable and controllable (**7**) and to demonstrate that raising the softening temperature of this very high purity copper from below 140C to well over 300C might be accomplished with thousandths, or at most a few hundredths, of one per cent of less costly metals. Antimony at about 0.006 or 0.007 per cent, tellurium at 0.005 per cent, selenium at 0.010 per cent, phosphorus at 0.020 per cent, tin at 0.010 per cent or cadmium at about the same level—each could contribute the desired high softening temperature without significantly impairing that property—conductivity (**8, 9**). One may properly ask when we shall expect an effect on practice and on specifications. To be sure, the presence of oxygen in these "alloys," if they may properly be so designated, is important in relation to both properties and fabrication (**9**), but the significance lies in the demonstration that the job can be done and additions once regarded as of indifferent or damaging propensities may now be usefully employed. Such are the upsets that result from progress in science and technology.

Whether it precisely fits the declared scope of this review may be fittingly questioned, but a statement on the deoxidation of copper and copper-rich alloys need not be lengthy. The metal has been effectively deoxided by phosphorus and by calcium-boron alloy, the additions amounting to a few hundredths of one per cent and the reduction in conductivity being very small if there is perfect control of the reaction so that residuals are limited to a few thousandths. Lean boron-copper alloys also are used to introduce boron but without any significant amount of any other element, the boron added being one or two hundredths per cent and the residual in this case also a few thousandths; added alone in this manner the impairment to conductivity is extremely slight in view of the very low solubility of the element, the remainder believed to exist as a fine dispersion of boride (**10**); thus the required control may be less exacting. Lithium also is used, either as a lean lithium-copper alloy or a rich lithium-calcium alloy (**11**). The amounts are of similar magnitude as for a boron addition, and of course the copper alloy requires less control than the calcium alloy in view of greater weight and no danger due to solubility of an element definitely lowering conductivity (calcium). Where this property is not a major factor one finds copper with 1 per cent cadmium finally deoxidized with lithium-calcium alloy (**12**).

Similar processing is applied to some bronzes such as those containing both tin and nickel and to other copper-rich alloys that require deoxidation. Phosphorus is most widely used, economy being an important consideration, but principally in finished products not requiring an approach to maximum conductivity and in which, therefore, control is simple and residuals may be several hundredths of one per cent.

Phosphorus appears again as a benefactor in the treatment of a related alloy, yellow brass, with a claimed result of grain size reduction and a favorable effect in the control of season cracking (**12**). The latter phenomenon, frequently a cause of grave concern, accepted now as one manifestation of what is generically titled stress-corrosion, was a most common cause of failure in single-phase copper-zinc alloys. Equally disturbing, and also frequently encountered was that corrosion phenomenon of many zinc-rich copper-base alloys referred to as dezincification: the preferential solution or at least retention in solution of zinc when the alloys were exposed to certain corrodents, notably sea water. Strangely enough, with all the need for correction, a solution of the problem was known long before it was practically applied (**13**). As may be observed in the specifications of this Society, 0.03 to 0.04 per cent of either arsenic or antimony or phosphorus will provide immunity to Admiralty metal or aluminum brass condenser tubing, with arsenic appearing to be the producers' preference.

The aluminum industry has, during more than three decades, provided an increasing number of practical illustrations of benefits flowing from minute additions.

The earliest and most widely known of the group assumed almost immediate industrial importance and still enjoys extensive application. It is, of course, the aluminum casting alloy containing 13 per cent silicon, modified by the addition of a few hundredths of one per cent sodium (**14**) (potassium may be used but is naturally more costly and is technically no more advantageous). The addition of sodium is made just prior to casting into molds, and while it should, for the optimum effect upon mechanical properties, be varied in amount according to the sodium content of the molten alloy and the cooling rate of the castings, it is ordinarily close to 0.05 per cent. The retention of about 0.010 per cent sodium with a microstructure of fine and well-dispersed silicon particles without appreciable coarse dendrites of solid solution or large silicon crystallites insures that the "modified" alloy will possess a tensile strength about 35 per cent higher than the "normal" alloy along with a fourfold increase in ductility as measured by elongation in the tension test. It has long been known that this "modification" by sodium was the result of undercooling since the eutectic temperature is effectively lowered (13C) and, the liquidus line remaining unchanged in direction, the eutectic composition is appreciably raised (2.4 per cent). It is the latter silicon content that must be closely approached but not exceeded for best results (**15**). More than one view is held, however, of the mechanism producing this undercooling. With the solubility of sodium in aluminum being less than 0.003 per cent at the solidus temperature of the binary system (**16**) and the likelihood of this low value not being increased by alloying with silicon, the view that sodium precipitates from the melt and nucleates, or interferes by adsorption with the growth of silicon crystallites, commands attention. Quite recently however, there has appeared what purports to be proof of the existence of an aluminum-silicon-sodium compound and the claim that it serves as the growth-controlling agent (**17**). The development of both strength and toughness in an alloy having casting characteristics that eminently qualify it for objects of very large area and thin sections was a major contribution to metallurgical technology.

Another example, and one of quite intriguing character, deals with the influence of small amounts of tin on aluminum-copper alloys, containing additionally some manganese, a little or several per cent of silicon, with or without nickel (18). Specifically, an alloy of the duralumin variety (4.4 per cent copper, 0.8 per cent manganese, 0.8 per cent silicon) with a small addition of tin will develop, on quenching and aging, the customary 65,000 psi tensile strength but with the high yield ratio of over 80 per cent. The tin addition may be as little as 0.005 per cent, although a few hundredths are preferred. Magnesium as an impurity (it is strangely enough in larger percentage one of the original components of duralumin) is detrimental to this high yield ratio and must be held to less than 0.005 per cent for best mechanical properties. But if contamination does occur and the magnesium content rises to a few hundredths of one per cent, as little as 0.05 per cent cadmium added will restore the original properties (19).

In the production and processing of aluminum conductor wire small amounts of the metals chromium, titanium, zirconium, vanadium, and molybdenum have a detrimental effect upon conductivity; when these metals are present to the extent of a few hundredths of one per cent, adding boron in the same order of magnitude largely negates the loss in conductivity (20). Similar boron additions also render unnecessary the customary annealing of severely cold-drawn aluminum wire since the conductivity in the presence of this additive will be equally high in the hard condition with the accompanying higher strength (21).

Alloys of aluminum containing magnesium (alone or with other alloying metals) are greatly benefitted by including in the composition very small percentages of beryllium. This is not a contribution to mechanical or physical properties but is in the nature of a chemical change. In solution heat treatment or in heating for fabrication at temperatures above 800 F, the discoloration and blistering (not the type of blistering due to release of internal gas on annealing) that occasionally ruins finished or semifinished articles may be avoided by using a chloride atmosphere and adding to the metal from a few thousandths to a few hundredths of one per cent beryllium (22). The same metal, in the range of about 0.003 to 0.010 per cent, added to this same group of alloys inhibits oxidation of their surface when molten, preventing loss of magnesium and the formation of objectionable dross without the use of fluxes or inert atmospheres (23). In the die-casting process with metal maintained in the molten state for long periods, the advantage is an obvious one.

Boron has also benefitted these aluminum-rich aluminum-magnesium alloys. With the heat-treatable 10 per cent magnesium type, with or without a quarter per cent beryllium addition, bubbling through the melt of boron trichloride for a time sufficient to leave a residual of about 0.010 per cent boron effects grain refinement, simultaneously raising the tensile strength some 15 to 20 per cent and the elongation about 40 per cent (24).

These are just a selection from many references to the use of minor additions to aluminum and its alloys. Prominent among this extensive group, one finds methods for the control of macrostructure and of other properties but especially of grain size, including in addition to the well-known and widely-employed application of titanium in somewhat larger quantities (of the order of 0.10 per cent) (25) reinforcement of the titanium effect by much smaller additions of beryllium and boron (25), boron alone (25, 27) or supplementing larger beryllium additions (27), columbium or tantalum in very small amounts (**28**) and with great frequency the element calcium (**29, 30, 31**). This list makes no pretense of completeness.

With magnesium-rich alloys suffering in far greater measure from loss by volatilization and oxidation when molten, and the early need for preventives including fluxes, inert gas atmospheres, and protective additions to molding sands, it would be natural to anticipate protection to be afforded in a similar manner to that functioning for aluminium-rich alloys containing several per cent of magnesium. However, beryllium added in like amount appears to be an overdose for this purpose while at the same time detrimental in other respects, serving in some instances only to coarsen the grain, reducing tensile strength and elongation by amounts such as 10 and 25 per cent, respectively. Quite contrary to expectation, protection against volatilization and drossing may be obtained in these alloys with only one tenth as much beryllium added. One finds the claim that as little as $\frac{1}{2}$ part per million is effective (**32**).

There are statements in the literature, however, that appear most contradictory. One such is to the effect that 0.025 per cent beryllium and 0.05 per cent zirconium impart to magnesium-base alloys grain refinement and resistance to oxidation (**33**). If at a lower level beryllium produces grain coarsening but in larger amount effects refinement or cooperates with this small amount of zirconium in producing such a result, the specific conditions should be determined, for elsewhere one notes that the zirconium requirement is very much higher, usually about three quarters of one per cent, to serve the same purpose (**34, 36**). Some careful experimentation and publication of the results are required.

This problem of grain refinement in magnesium-base alloys and its beneficial effect upon mechanical properties is a most interesting one and many solutions have been proposed. Among them may be mentioned adding a few thousandths of one per cent of aluminum and a few hundredths of calcium followed by bubbling of nitrogen through the melt, employed with 2 per cent manganese alloys (**35**). In alloys containing 3 per cent zinc, grain refinement is produced by zirconium (only that which is acid soluble is stated to be effective and must be at least 0.3 per cent) (**36**), but the grain dimensions may be reduced to one half or less by a few hundredths per cent of barium or strontium or both (**37**). Yield strength is raised thereby an additional 15 to 20 per cent. Unfortunately, molten alloys so treated oxidize readily, so that use of the method would necessarily be limited. Another solution, devoid of this limitation, is the addition of a few hundredths per cent beryllium and three quarters per cent zirconium, with introduction into the magnesium simplified by prealloying both metals with silver (**34**).

Calcium in very small amounts has also been added to supply benefits to some magnesium alloys (**38**); likewise the rare earth elements (**39**).

Small additions are not strangers to the metallurgy of zinc. One of the first of the major metals of industry to be available in high purity (99.99+ per cent), it is certain that the corrosion problems of zinc-base die castings would otherwise have remained unsolved for a much longer period. It is now well known that 0.03 to 0.04 per cent magnesium in zinc die castings alloyed with aluminum (or aluminum and copper), which are preferred for their minimal attack in the molten state on steel dies, will prevent intercrystalline corrosion and growth during normal use (**40**). It is imperative, however, that the soft, low-melting, heavy metals—tin, lead, bismuth, and cadmium—be practically absent. On the other hand, in a rolled zinc alloy containing usually about 1 per cent copper, magnesium performs quite a different function, namely, adding creep resistance. Curiously enough, for this application, traces of aluminum are very damaging and traces of the above heavy metals not at all disturbing (**41**). With the work on die castings so painstakingly accomplished within the Society, only the need for an approach to completeness justifies its mention here.

With several alloying metals, lead forms precipitation-hardenable systems of considerable interest, at least part of that interest centering upon the combined effects of retardation of recrystallization in the alloyed matrix and precipitation of the second phase. Addition of 0.005 per cent silver to lead of 99.998+ per cent purity (the limit of room temperature solubility) makes possible an increase of 16 to 17 per cent in tensile strength, entirely the stiffening of the solid solution; doubling the addition to secure precipitation as well produces about 20 to 21 per cent strength increase. Calcium as the precipitation-hardening agent is much more effective, no doubt the result of the comparative strength and other properties of the precipitate. At the limit of solid solubility at room temperature (0.010 per cent calcium) strength is raised only 11 to 12 per cent (the low value when compared with the similar effect of silver may be due to shorter aging time), but with two and a half times this calcium content the increase rises to about 115 per cent—and with further additions may be caused to multiply almost fourfold. Precipitation hardening in a lead alloy with 1 per cent antimony may be effected by the addition of only 0.005 per cent arsenic, strength being increased after quenching and aging anywhere from about 40 to about 70 per cent depending on the time of aging (**42**). For cable sheathing, this result is undesirable, but who will say that for another use the property will not some day prove advantageous.

A phenomenon not at all clearly understood is the increase in strength of 99.998+ per cent lead by the addition of 0.005 per cent nickel amounting to about 9 per cent, contrasted with the same addition to 99.98+ per cent lead causing a tensile strength increase of over 40 per cent (**42**). Here is another instance of the need for ultrapure metals as a basis for a true understanding of behavior. Someone will surely unfold this relationship between traces of nickel and traces of silver, copper, bismuth, antimony, arsenic, zinc, and tin in a lead matrix.

Perhaps part or all of the improvement may be the combined effect of some of these impurities on grain size. However, not only the silver and the calcium just noted refine the grain of lead in the course of their contributions to strength, for it has been recorded that a few hundredths per cent of selenium or tellurium or copper will produce similar results and 0.01 to 0.02 per cent lithium will cause extremely fine grain (**43**). Grain refining would seem to be an attribute of many metals in solid solution in lead.

Interesting effects are produced in cast monel metal of 30 to 32 per cent copper content (its sulfur controlled by a suitable addition of magnesium) on adding small quantities of boron. With carbon content high (over 0.2 per cent) due to low silicon or other causes, one to two hundredths per cent boron promotes graphite formation, greatly reducing both strength and ductility. But if carbon is low in the usual 1.5 per cent silicon alloy, short-time tensile strength at 1000 F is raised more than 50 per cent and ductility trebled or more; simultaneously the boron reduces the detrimental effects resulting from the presence of lead. A more certain method of reaching the same end, however, appears to lie in the introduction of 0.05 per cent or slightly more of zirconium (**44**).

An equally complex metallurgical situation has been found in efforts to improve the life of nickel-chromium alloys exposed to high temperature such as the 80–20 wires and grids used for electrical heating elements. Actually two solutions have resulted. In one the additions consist of a few hundredths per cent alkaline earth metal, preferably calcium, a similar quantity of phosphorus or arsenic, preferably the latter, along with 0.15 to 0.20 per cent of rare earth metals, preferably cerium. The other solution comprises the introduction of calcium, 0.03 per cent residual preferred, thorium with a preference of 0.07 per cent and a few hundredths per cent of phosphorus, arsenic, or antimony with arsenic preferred (**45**). These deoxidation and alloying procedures likewise improve the life of the 65 per cent nickel, 15 per cent chromium, 20 per cent iron alloys, while the second method is recommended also for the 20 per cent chromium, 5 per cent aluminum, 75 per cent iron resistance alloy. Importance attaches to these developments in view of raising the service life in the useful temperature range of each type from two to fourfold, which is a striking alteration by such small additions to alloys already capable of forming oxide films of great tenacity, ductility, and imperviousness.

The essentials now recognized for less than five years and greatly increasing the crying need for perfect purity, the semimetals or semiconductors silicon and germanium have excited wonder and curiosity (**42, 46**). The methods employed in their preparation and treatment are the methods of the metallurgist so that discussion here is not improperly placed.

It is difficult to describe briefly these products and the physical mechanisms responsible for their functioning. With the conductivity of these elements several orders of magnitude below that of the metals, the problem has been to reach an intermediate but rigidly controllable constant level. The conductivity of silicon is improved about 60 fold by 0.001 per cent boron and 360 fold by 0.01 per cent. Phosphorus at 0.0029 per cent raises conductivity 100 times and with double this addition, over 300 times. These two elements as selections from Groups III and V of the periodic system contribute to the movement of electrical charges by boron being a donor of electrons to the silicon and phosphorus an acceptor of electrons from the silicon. But, both elements segregate even in small ingots, although in different manner. The problem is to so balance the amounts of these two "alloying" elements and to so control the solidification of the silicon ingots that a product of uniform resistivity may be obtained. This has been done. With germanium, a similar balance of Group III and Group V elements is sought with not only segregation but an additional variable—heat treatment—to be included.

In actual use it appears that the approximate additions of boron to silicon are several parts per million and of antimony to germanium are a few parts per hundred million. Obviously, for perfect control, purity of the basis elements must approach perfection and the attack on this problem grows ever more intense.

Interference with grain growth in the precious metals by incorporation of finely dispersed oxides may be of questionable propriety in this presentation, as the amounts of thoria employed with platinum (earlier with tungsten) and more recently platinum alloyed with small percentages of tungsten have ranged close to one half or three quarters of one per cent. The use is an important one since the structure of the articles, normally made by powder metallurgical methods and subsequently hot and cold worked, persists in fabrication and use at temperatures of 1800 F and higher (47). The temptation to mention this subject comes from publication of other methods, including instead of pressing and sintering the melting of platinum or platinum group metals or their alloys, adding in a nonoxidizing atmosphere and in the absence of a flux, oxide-forming metal such as silicon or beryllium or thorium, changing the atmosphere to an oxidizing one for a brief time, and solidifying, thus obtaining a fine dispersion of grain-controlling oxide. The percentage additions may be much smaller than customary (48). The principle has been employed also with nickel and with copper (47). Further inducement is presented by another practice, in this case applied to high-purity silver. The metal, alloyed with a few hundredths per cent of aluminum or magnesium is annealed in air and cooled; aluminum oxide or magnesium oxide serves as a hardening agent, forming *in situ* in accordance with the extent of penetration of oxygen on heating and precipitating on cooling. The silver is thus case hardened. Cases of very considerable hardness may be obtained by more richly alloying with these metals. A similar effect, but with less hardness resulting, may be secured with zinc, or in the presence of a few hundredths of one per cent of iron by exposure to zinc vapor in a hydrogen atmosphere (49).

Tin seems now also to be in the realm of improvement by minor additions of other metals. There are indications that a few hundredths or even a few thousandths of one per cent of some metals may prevent or long delay the transformation at low temperatures of normal tin to the gray pulverulent variety. Recent experiments in coating with tin-rich alloys indicate the likelihood that very small amounts of aluminum, silver, and more especially cadmium in the coating may serve advantageously (50, 51).

Thus many metals are most interestingly altered by the introduction of these very small amounts of other elements. Among the new arrivals on the industrial scene in rather pure form and of good ductility are titanium and zirconium. Control of oxygen, nitrogen, and carbon within narrow limits and in the order of hundredths of one per cent will make possible harder, though naturally somewhat less ductile, forms for uses requiring special qualities. Indications are that the still more recent arrivals—ductile chromium and ductile vanadium—will respond to like treatment.

Having given consideration to the various means of deoxidizing copper and its alloys, it seems only logical to note the deoxidation of steel by aluminum, leaving a residue of combined metal and oxide in the range of about 0.01 to 0.04 per cent aluminum with grain refinement resulting at the higher levels and dependent somewhat on carbon content. In similar quantity calcium is being employed in special steels, again primarily for combination with oxygen. Most probably for sulfur control, there is widespread use, in the more highly alloyed austenitic corrosion-resisting steels, of cerium in slightly larger dosages and (possibly for altering the form of the nitrogen present) of the complex boron alloys to the extent of a few thousandths of one per cent boron; in some cases combinations of these two additions have proven particularly efficacious in reducing losses due to shortness experienced in hot working.

I cannot, of course, continue a discussion of small additions to steels without making mention of vanadium. One finds additions to the extent of 0.02 to 0.05 per cent in carbon and low-alloy tool steels to contribute temperature-stability and small particle size to the carbide phase. Similar amounts are added to heavy forgings to insure adequate yield strength with maintenance of the maximum ductility, especially in transverse and radial directions. These small percentages are added also to low-carbon medium-manganese plate in association with minute amounts of titanium, and in a number of constructional low-alloy steels where, at about 0.04 per cent vanadium, the element contributes a maximum to hardenability. For reasons of yield strength and ductility, it is used in many steel castings, although at slightly higher levels of addition.

Cast iron metallurgy also has advanced through the medium of these small additions. For close to three decades addition of calcium as calcium-silicon alloy to molten iron of suitable composition, with the retention in the iron of about 0.01 per cent of calcium, has provided consumers with metal having up to 60,000 psi tensile strength and actual tensile ductility of the order of $\frac{1}{2}$ per cent, the result of very small graphite flakes in random arrangement (52). The change of size and distribution has permitted the matrix to approach full development of its inherent strength.

Some use has been made of the chilling effect in cast iron of a few hundredths per cent of tellurium (53).

Now the industry is in the throes of a revolution, undoubtedly to be an extensive and most important one. The literature is already impressive. With the initial discovery that 0.02 per cent of cerium (combined with a late inoculating addition of 0.50 per cent or more of silicon) could cause the graphite to precipitate in spheroids instead of as flakes (54) and the subsequent discovery that the same structure resulted from the addition of magnesium to yield in the finished iron about 0.04 to 0.08 per cent (55), came a new kind of cast iron. As cast or suitably heat treated or mildly alloyed the properties range from 60,000 to 120,000 psi tensile strength and from over 20 to about 4 per cent elongation. This new cast iron product is already making inroads into other fields of ferrous metallurgy and will continue to do so. The discoveries have given rise to numerous alloys for the introduction of these elements, efforts to effect reduction of compounds of the elements in contact with the iron, studies to avoid the need for final inoculation, studies of alloying and of heat treatment of the product, and many others. From all this research it appears likely that the amounts of these additions may one day be reduced so that, for example, instead of adding 0.15 to 0.35 per cent magnesium in any selected form to yield a recovery of the above 0.04 to 0.08 per cent, much smaller additions may be made and the full effect obtained with retention of only 0.010 or 0.015 per cent magnesium with or without very reduced amounts of cerium or other suitable metals. As with many other large effects due to small additions to metals, no generally acceptable mechanism has yet been proposed.

There has been more discussion in recent years of the use of boron-containing alloys in steels than of any of the other minute additions that have been presented thus far. In part this is due to their importance in the country's economy and in part to the very large number of persons engaged in their production, processing, and use. Individual ideas are expressed as to their merits, as well as the manufacturing methods and the probable future of these steels, but there can be no denial that tons upon tons are each day being converted into finished load-carrying parts in appliances, machines and heavy equipment.

Historically, while seeking a method for the effective use of small additions of vanadium to intermediate manganese steels, and having determined to study thoroughly the possibilities of association with titanium, and having further made a series of titanium-containing alloys by aluminothermic reduction so that they contained aluminum residuals, an opportunity was afforded to try the alloys in not only medium-manganese but also in carbon steels. The results exceeded expectations. There followed rapid and extensive experimentation with alloy compositions and production methods, with trials in steels, with testing methods and all else. Early it was found that the judicious use of ferro-boron would occasionally yield the same steel properties; only later was it learned that the original alloys due to the raw materials used and process employed contained small amounts of boron (**56**). It was then established that vanadium was a most important additive but not an absolute necessity. This beginning was in 1937.[4]

Only in 1944 was acceptance becoming substantial. By 1950 it was of major magnitude—one more instance of slowness in adoption of a new material or a new technique.

Much work followed the early trials, seeking to improve the alloys and their method of use and to clarify the cause of their behavior (**57**). No generally accepted explanation of the mechanism of the observed phenomena has yet appeared, but the alloy accidentally yet fortuitously prepared for that first commercial heat of steel with no more than 5 parts per million of boron added, still is in good repute and retains a substantial share of the market while the complex alloy without vanadium put into production just a few years later retains the maximum. In the sixteen years since I had the thrill of contributing to that first steel experiment, a voluminous literature has appeared concerning these steels and their properties—most of it during the past six years—and it still continues its rapid growth.

The initial observations came at a time when a common method of evaluating quenched-and-tempered steels in not too large sections was by simple formulae embodying predetermined relative weighting of tensile strength and reduction of area in the tension test. By this criterion, the first steels had "merit indices" equal to the maximum or higher than had been previously observed with consistency in low-alloy steels. Later came the dependence upon various means of measuring hardness penetration on quenching, culminating in the Jominy hardenability method. Measurements using this principle indicated for the boron-containing steels properties of such significance, and its use to control subsequent substitutions of these new steels for the older and richer alloy steels so greatly concerned both steel-makers and users, that the hardenability contribution by the boron alloys has far outweighed in interest all other properties. When comparing these boron-containing steels and their lower content of customary alloying elements with the richer alloy steels that they replace, full consideration should be given to their advantages in hot- and cold-working and in numerous machining operations. Except when these steels are in their ultimately hardened-and-tempered condition, they are much more readily worked and the greatly prolonged life of tools and dies used to fabricate them as well as the easier heat treatment preparatory to such fabrication should not be brushed aside.

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[4] Reference continues to be made to the mention of the use of 0.001 to 0.1 per cent boron to steels in U. S. Patent No. 1,519,388 of December 16, 1924, filed August 13, 1921. It will be clear from what follows that 0.001 per cent boron could not contribute significant hardenability, if any at all, if it were not previously alloyed with titanium, and there is no mention of this in the patent. The statement appears that 0.001 per cent boron "revolutionizes the structure" in respect to "fineness of the texture"; also that 0.002 per cent boron produces in a steel of 0.2 per cent phosphorus [*sic*] "a texture of such fineness as cannot be obtained in phosphorus-free steel." On grain size, the effect of boron alone is quite the opposite. Hot-shortness is stated to appear only at more than ten times the actual known level. The patentee's knowledge of the behavior of boron-containing steels is qualitatively and quantitatively completely wrong, opening the entire paper to question.

As previously mentioned, there is no general acceptance of the ideas or theories put forth to explain the mechanism of behavior of these boron alloys. But certain practical aspects of the alloys and their method of use warrant consideration regardless of whether one subscribes to:

(*a*) The concept that concentration of the boron at austenite grain boundaries as an interstitial iron-boron solid solution, the presence of which lowers the rate of grain-boundary nucleation, is the cause of higher hardenability (**58**), or

(*b*) The boron, or portion thereof in solution at time of quenching (irrespective of its location), decreases the rate of nucleation of ferrite and carbide in the vicinity of the nose of the *S*-curve, thereby contributing to martensite formation (that is, increasing hardenability), or

(*c*) Acid-soluble nitride nuclei (principally aluminum nitride) are reacted upon by boron with the formation of relatively inert boron nitride, thus decreasing nucleation, so that in the complete absence of nitrogen (and possibly, aluminum) boron in these small amounts would not display this influence because hardenability would be high initially (**60**), or

(*d*) An expressed but not yet detailed order-disorder phenomenon or an equally incomplete exposé of restraint of nucleation by patchy grain boundary films (**61**).

Within a relatively short time following the initial discovery, the making and testing of hundreds of alloys demonstrated conclusively the fortuitous composition of the first alloy produced and tried. It was clear that after effective aluminum deoxidation of the steel, a little aluminum in the alloy served to keep any subsequently introduced oxygen from reacting with the remaining alloy components; its titanium (or in part zirconium) content was then available for combination with nitrogen, leaving the boron to perform its function by whatever means the future may disclose. The key combination is *titanium-boron in the added alloy*; it has been proved by every subsequent commercial development, and this explanation recently was confirmed by British experimentation. In the absence of titanium, the required boron addition is three to fivefold regardless of preadditions of aluminum and separate preadditions of titanium or zirconium alloys. Presumably, part of the boron, by combining with oxygen or nitrogen or both, serves to protect the remaining boron so that the latter may be effective in the hardening process.

More specifically, in quenched-and-tempered steels the full hardenability and accompanying effects may be secured, following aluminum deoxidation, by the addition of 0.001 per cent boron or slightly less, in the form of an alloy of the type just described. By other means, at least 0.003 per cent boron must be added, and even then reproducibility suffers. If sufficient vanadium is present in an alloy of the preferred type the boron addition need be only 0.0005 per cent or slightly less—that is, only five parts per million!

For several years, intense research has been under way in many laboratories, here and abroad, but as yet no generally satisfying and accepted explanation has been forthcoming. The number of observations that such an explanation must fit may be a partial cause for present skepticism since one must include alloy behavior, response of different steels made by different practices and of differing oxygen, nitrogen, and carbon contents prior to the addition of the boron alloy, loss in hardenability on exposure to high temperatures, and others. The explanation must satisfy the behavior of quenched and tempered steels and also as-rolled or normalized low-carbon steels to say nothing of the effects of other alloy content. We are well aware not only of the occurrence of the boron in more than one form, but also of the change in the state of combination of the nitrogen on adding these boron alloys to steel. It has been possible to recognize by a prescribed analytical method whether or not the addition has been completely effective. However, the limits that may be applied to the results vary with certain practices, including the type of alloy. Research is needed into the meaning of such data before the question of boron behavior can be fully clarified. To him who opens this door should come rich rewards. I subscribe to a recently expressed view that whoever cracks the secret may point the way to a new metallurgical field of much broader scope than just that of the immediate problem.

Having now trodden completely across the metallurgical horizon, I return to the subject matter of Dr. Gillett's last "critical review (62)." In doing so I must depart, though ever so slightly, from the declared purpose of *this* review and refer not to a minute "content" of one element in another but a minute "difference" which is actually an "addition" and should thus qualify for inclusion in this presentation. You will recall that that last paper to which I have previously referred dealt with very-low-carbon stainless steels of the austenitic type. Under the title "Present Knowledge of Low-Carbon 18-8" one will find the usual healthy criticism and the effects of keen perception. I quote:

> "Obviously the lower the carbon, the less the difficulty. Just how low it is necessary to go has been a question. It can even now be answered only when we are told just how the material will be used."

Again, after considering all the effects of processing and composition variables, including the disagreement as to the maximum limits for both carbon and nitrogen to insure immunity from grain-boundary precipitation of a corrosion-promoting phase, plus the voluminous data on corrosion rates following many variations in "sensitization" procedure contrasted with the limited data on structures assumed to embody more or less the conditions employed in these tests, I quote again:

> "Why do we have so much indirect and so little direct evidence on so vital a point?"

Then, by way of characteristic emphasis:

> "From the point of view of the materials engineer, the condition is "snafu" because of indirect tests and the uncertainty of their translation into actual service."

Always the scientific method but always combined with the practical outlook.

During the past decade or slightly more there has been rapidly growing production and use of these very-low-carbon austenitic chromium-nickel steels. Before this all began, but much more vigorously during the growth period there have been substantial sums expended in studies of the steel-making methods and the properties and fields of application of the product. The objective, of course, has been to circumvent the use of correctives for intergranular corrosion. Austenitic chromium-nickel steels of similar chemical composition but of higher carbon content (0.08 per cent maximum or more), if not protected by the incorporation of an adequate quantity of a carbide stabilizing element, may deteriorate rapidly in any one of many corrosive media, some very active, some slower in their disintegrating effects, after being subjected to certain thermal cycles such as those encountered during welding. Elimination of these stabilizers tends also to avoid loss in general corrosion resistance. Another driving force is the problem of availability. I refer, of course, to columbium and titanium and to a lesser extent to vanadium.

It appears that austenitic steels of the chromium-nickel type with a maximum carbon content of 0.03 per cent serve adequately without danger of deterioration due to intergranular corrosion, when subjected to any number of variants of the "sensitization" test or when welded or otherwise heated in fabrication in temperature zones that would produce more or less rapid failure, were the carbon higher and stabilizers absent; many corrodents encountered in commercial use are without significant effect. Two conditions are responsible for the steelmakers' ability to supply industry with substantial quantities of this steel. The first is the successful manufacture of very-low-carbon ferrochromiun with 0.03 or 0.02 per cent maximum carbon which has been made in considerable quantity in France for several years following production on a moderate scale for well over ten years, and may now be obtained in the United States as products of two distinctly different processes. The second is the rapid growth of the metallurgical application of oxygen and its consequent availability in large tonnage at low cost.

However, while striving for perfection in this transition from steels of 0.08 per cent maximum carbon content to the types of 0.03 per cent maximum, some opportunities appear to have been overlooked or given inadequate attention. Manufacturing practices differ not only in their major features but in their details in making these very-low-carbon steels. But in producing steels of an actual carbon content of 0.020 to 0.025 per cent (necessary to meet an 0.030 per cent maximum specification) certain features are common to all methods—the oxygen content of the bath is high, the temperature is high, and the time required for the lowering of each 0.01 per cent carbon is progressively longer. The net result is punishment to equipment, higher oxygen consumption, possibly altered properties (other than corrosion resistance), and greater chromium losses if stainless steel scrap or high-carbon ferrochromium are among the raw materials. Ultimately, these items must be reflected in costs. Since the actual carbon content of the steel in the 0.08 per cent maximum carbon grades has always been the basis for computing the required residual content of corrective carbide-forming element and since the actual 0.020 to 0.025 per cent carbon in the 0.03 per cent maximum carbon grade appears to serve current needs adequately, it seems necessary to present the following question: Would a steel specification at 0.04 per cent maximum carbon with a corrective applied to take care of only the excess of 0.01 per cent carbon present advantages?

The following appear likely:

1. Wear and tear on melting equipment greatly reduced. Ferrochromium available at 0.03 and 0.02 per cent maximum carbon means that the relief in melting the steel base can be greater than this 0.01 per cent specification difference, or chromium losses less where chromium-containing materials are in the charge.

2. General corrosion resistance of the steel will not suffer if the corrective is applied only to the excess carbon. This should be especially true if the nitrogen content of the steel bath is kept at or reduced to the lowest possible level; the oxygen content is automatically benefitted.

3. The higher carbon content may pro-

duce a change in the useful strength of the product as measured by its "elastic properties."

Steels of this suggested intermediate carbon have been made but apparently none to which this specific suggestion applies. Always they have been so-called fully corrected. But it is obvious from successful use of the very-low-carbon steels that this need not be done, that general corrosion resistance need not suffer, and that "elastic properties" may be benefitted. Current disagreement as to the limit of solubility of carbon in 18-8 is of no import since with 0.020 to 0.025 per cent actual carbon content, whether 0.010 or 0.020 per cent is soluble under equilibrium conditions can have little practical effect. Work along this line needs to be done. Unfortunately, perhaps only the first steps can be taken in the laboratory; none but commercial melts will provide a complete answer.

I have recited all this detail so that you may arrive, with me, at a realization of the great number of instances in which minute amounts of elements added to metals and alloys have brought improvements that have produced or may soon produce industrial benefits that add to man's well-being. All here recorded has occurred in one man's professional lifetime—a span of about forty years. It is, of course, quite certain that all of the known examples falling within the scope of the title have not been cited. It is equally certain that to set arbitrary limitations on the coverage in such a review may cause instances of great interest to be excluded; yet to a degree this had to be done for if, instead of dealing with hundredths, thousandths, and lesser percentages of addition, I had encompassed just one higher order of magnitude, the flood gates would have been opened, the boundaries would have become almost limitless, and you would have had not a one hour lecture but a volume or two.

Moreover the smaller additions display in so many cases more novelty and evoke more striking interest, for they present the more sensitive responses of metals to alloying or to correctives, whichever the case may be.

Consideration was given to expressing the quantities not as weight percentages but rather atomic percentages or even gram-molecules per unit of volume. The latter two methods have more meaning to the scientifically-inclined, especially with metals of such varied physical properties, but weight percentages are so common in specifications and in daily practice that they are in general more quickly grasped.

Scanning these citations of the aid given by single atoms to many thousands, there is no readily apparent basis for generalization, but one cannot escape the observation of some similarities among the elements imparting the sharp improvements in properties. Most of the observed instances result from the use of elements of low atomic weight and high chemical reactivity—lithium, beryllium, boron, sodium, magnesium, aluminum, calcium—all in one corner of the periodic system of the elements and comprised in groups I, II, and III. With certain elements with which they form compounds, heats of formation are high. They are in most cases active in their association with oxygen, nitrogen, even hydrogen, but also with sulfur and the halides.

A lesser number of instances are concerned with the addition of heavier elements, namely, titanium, zirconium, thorium in group IV, phosphorus, arsenic, antimony, and vanadium in group V. The rare earths must remain in their intermediate position. Among these heavier metals it is the elements of group IV which again display high heats of formation with certain other elements.

It is this situation that gives rise to the frequent mention of correctives. But obviously we cannot treat of them without consideration of the medium in which they operate.

Several times, mention has been made of the lack of adequate proof of assumed mechanisms by which these enhanced properties are secured and likewise the frequent long intervals between observation of an effect and recognition of the cause. I offer you now no hope of a simple explanation of such diverse phenomena as are here recorded, if for no other reason, only because of the great differences in the basic metals to say nothing of the varied properties that are affected. One needs to consider questions of liquid and solid solubility and changes with temperature, the resulting precipitation of quantitatively minor constituents and the particle size, properties, and distribution of the precipitates, more or less continuous grain boundary films and their removal or dispersion, rates of diffusion, and a host of other features of behavior. When explanatory simplification arrives, there seems more likelihood for it to develop along physical lines and to provide the possibility of mathematical expression; to date the closest recorded approach may have been made by those associated with the development of the semiconductors.

Nevertheless one over-all fact stands forth clearly. Despite all the progress, the purity of our metals and the sensitivity of our various means for the measurement of the magnitude of impurities are not yet adequate. Further advancement is required not solely for the purpose of experimentation although admittedly it will afford the only perfect means of learning how to perform the subsequent reincorporation under control of specific elements removed, either by themselves or with other elements. We customarily desire to use in a single application only some of the properties of metals and alloys; it is likely that these are not usually developed to the fullest extent and that directional control may be possible. For this, however, new measuring tools will be needed, for there will be new criteria of "purity," and control within extremely narrow limits will assume maximum importance.

Conventional analytical methods need scarcely be mentioned. Spectrography also in many cases has reached its limit of service in this cause. Physical methods of identification must be provided to detect the presence of specific impurities and to measure ever-decreasing magnitudes. Tracer techniques and magnetic and optical measurements, many appearing of indirect approach, are beginning to assume importance. This may seem to be peering into the future, but in actuality it is not so. We have come to talk in these alloying and impurity problems of parts per million. Be not surprised if very soon there is discussion in terms of parts per billion—or beyond.

To reinforce these contentions I shall add just two significant instances. In the copper-copper oxide rectifier it has long been known that the very best performance results from the use of a particular type of copper, namely, electrolytic produced from specific ores. The reason for it is unknown; the spectrograph has afforded no assistance (**63**). More recently a single lot of spectroscopically pure tin was not transformed even when inoculated with gray tin powder. That the result was very likely due to something other than very high purity was indicated by the fact that after heating in vacuum at 1000 C for a brief period this lot was transformable into the gray variety (**50**). No explanation has yet been forthcoming. It appears thus impossible without new knowledge based upon new techniques to answere many of these metallurgical riddles.

This brings me to the end of my story. Only part represents personal experience. Hence it would have been most incomplete in the telling were it not for the writings of those who have so thoroughly investigated many of its aspects. To the records they have left and to many conversations with the individuals themselves, I am deeply indebted and now render due acknowledgment.

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PROPERTIES AND BEHAVIOR OF A HIGH-STRENGTH RIVET STEEL[*]

BY FRANK BARON[1] AND EDWARD W. LARSON, JR.[1]

SYNOPSIS

A high-strength rivet steel, developed by the Bethlehem Steel Co., which has certain advantages over other rivet steels meeting the requirements of the ASTM Specification A 195 – 52 T[2] is reported upon. The high-strength rivet steel is as ductile and is driven as easily and quickly as ordinary carbon rivet steel. At short lengths of grip, the average clamping stresses of the high-strength steel rivets are about the same as those of carbon steel rivets. However, at grip lengths of 2 in. and greater, the average clamping stresses of the high-strength steel rivets are appreciably greater than those of carbon steel rivets.

The behavior in static tension and in fatigue of butt joints fastened with rivets of the high-strength low-alloy steel was compared to that of joints fastened with rivets of ordinary carbon steel. For static loads, the efficiencies of the joints were about the same irrespective of the kind of rivet steel. The fatigue strengths of joints having a $1\frac{5}{16}$-in. length of grip were about the same irrespective of the kind of rivet steel. However, at longer lengths of grip, the fatigue strengths of the joints fastened with the high-strength rivets were greater than for similar joints fastened with ordinary carbon steel rivets. It was also observed that a decrease in the ratio of tension to shear of riveted joints can result in a decrease in the fatigue strengths of such joints.

The use of a high-strength rivet steel in connection with carbon, silicon, or other high-strength structural steels is at times of interest to the structural designer. In certain cases the substitution of high-strength rivets for carbon rivets permits a reduction to be made in the number of rivets required in a joint or splice. The substitution can affect the sizes of splices or of gusset plates. Objections are frequently raised to the use of high-strength rivets because of the high cost and difficulty of driving such rivets. In certain cases the rivets may meet the strength requirements but may be deficient in ductility and in impact strength after driving. Additional objections may exist due to the degree of hole filling and the small values of clamping force usually obtained for such rivets. A small clamping force and poor hole filling may permit an appreciable slippage of joints to occur at small values of load. A small clamping force may also result in lower fatigue strengths of joints fastened with such rivets than for joints fastened with carbon steel rivets.

Among the various high-strength rivet steels available may be listed the commonly designated silicon, silico-manga-

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[2] Tentative Specification for High-Strength Structural Rivet Steel (A 195 – 52 T), 1952 Book of ASTM Standards, Part 1, p. 564.

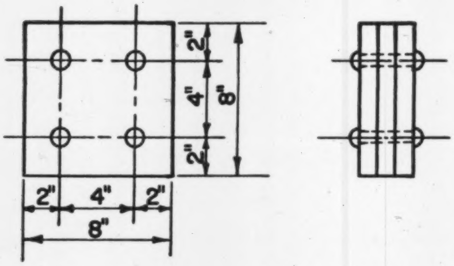
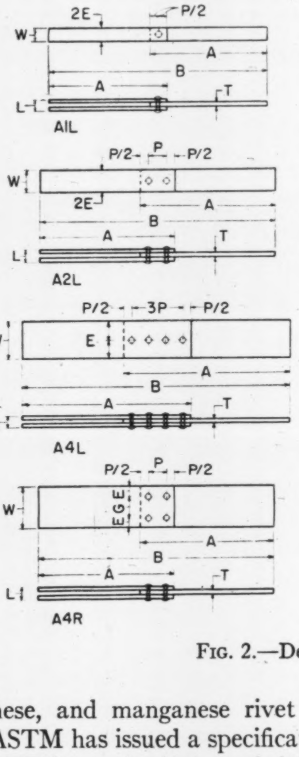


FIG. 1.—Dimensions and Details of Sample Specimens with Driven Rivets (Groups I and II).

Rivets: Carbon steel, ASTM Specification A 141, $\frac{3}{4}$-in. diameter
Alloy steel, ASTM Specification A 195, $\frac{3}{4}$-in. diameter
Holes: Drilled $\frac{13}{16}$-in. diameter
Lengths of grip: Ranged from $\frac{3}{4}$ to $4\frac{1}{16}$ in.



Details and Dimensions of Spesimens

Spec No.	Rivet Diam D in.	Dimensions, in.										Areas, sq in.		
		A	B	E	G	W	P	T	L	E/D	G/D	Tension	Shear	Bearing
A1L6	3/4	21	$38\frac{1}{2}$	$1\frac{3}{32}$		$2\frac{3}{16}$	$3\frac{1}{2}$	$\frac{13}{16}$	$1\frac{13}{16}$	146		1118	0884	0610
A2L6	3/4	24	41	$1\frac{25}{32}$		$3\frac{9}{16}$	$3\frac{1}{2}$	$\frac{13}{16}$	$1\frac{13}{16}$	2 38		2.21	1.77	1.23
A4L6	3/4	30	46	$3\frac{5}{32}$		$6\frac{5}{16}$	$3\frac{1}{2}$	$\frac{13}{16}$	$1\frac{13}{16}$	4 20		447	354	244
A4R6	3/4	24	41	$1\frac{3}{4}$	$3\frac{5}{8}$	$7\frac{1}{8}$	$3\frac{1}{2}$	$\frac{13}{16}$	$1\frac{13}{16}$	233	483	447	354	244
A2L7	7/8	25	43	$2\frac{1}{16}$		$4\frac{1}{8}$	$3\frac{1}{2}$	$\frac{15}{16}$	$1\frac{15}{16}$	236		299	240	164
A4R7	7/8	25	43	2	$4\frac{1}{4}$	$8\frac{1}{4}$	$3\frac{1}{2}$	$\frac{15}{16}$	$1\frac{15}{16}$	228	486	598	480	328
A2L8	1	25	43	$2\frac{11}{32}$		$4\frac{11}{16}$	$3\frac{1}{2}$	$1\frac{1}{16}$	$2\frac{3}{16}$	234		386	312	212
A4R8	1	25	43	$2\frac{1}{4}$	$4\frac{15}{16}$	$9\frac{7}{16}$	$3\frac{1}{2}$	$1\frac{1}{16}$	$2\frac{3}{16}$	225	494	778	624	425

Rivet Holes were Punched
Diameters of Rivet Holes 1/16 in. Larger than Nominal Diameters of Rivet
Approximate Tension : Shear : Bearing Ratio = 1.00 : 1.25 : 1.80
Plate Material, ASTM A7

FIG. 2.—Details and Dimensions of Joints for Series A.

nese, and manganese rivet steels. The ASTM has issued a specification A 195 – 52 T for such rivet steels.[2] This specification is based on the results of tests conducted by the U. S. Navy and those reported by Jonathan Jones (1).[3] The new rivet steel reported upon herein meets these specifications and has certain characteristics desirable in a high-strength rivet steel. The chemical composition of this new rivet steel is given in Table VII.

A program of tests was defined to determine the physical properties of the alloy steel rivets and the behavior in static tension and in fatigue of joints fastened with such rivets. Two groups of sample specimens and four series of butt joints were fabricated for the program reported herein. The clamping and physical properties of the alloy steel rivets and of carbon steel rivets were determined for several lengths of grip. A series of butt joints fastened with the alloy steel rivets was tested in static tension to determine whether a ratio of tension to shear of about 1.00 to 1.25 was satisfactory for such joints. Three series of butt joints were tested in fatigue to determine the effect of grip and clamping force on the fatigue strength of joints fastened with the alloy steel rivets and of joints fastened with carbon steel rivets.

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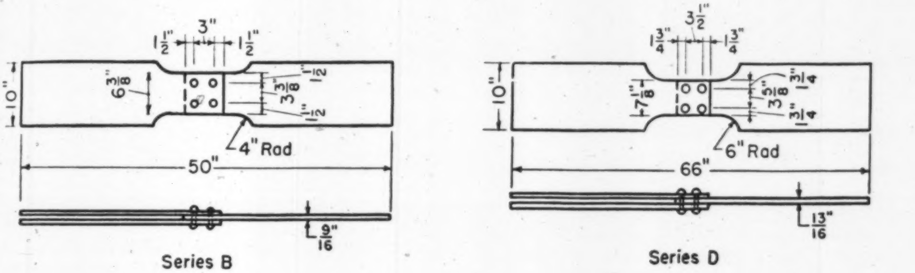
[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 609.

DESCRIPTION OF SPECIMENS

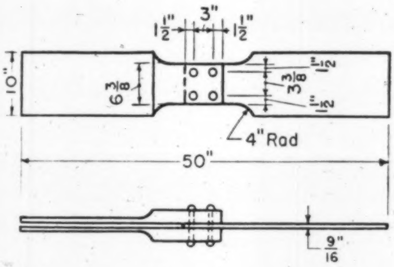
Two groups of sample specimens were used for determining the clamping stresses of the rivets. The dimensions and details of the sample specimens are given in Fig. 1. Each group was driven at a different time and included sample specimens fabricated with carbon steel rivets and with the alloy steel rivets. The rivets had a $\frac{3}{4}$-in. nominal diameter and were driven with a pneumatic hand-hammer in accordance with standard shop practice. The driving temperature for both kinds of rivet steel was 2150 F. Several lengths of grip, ranging from $\frac{3}{4}$ to $4\frac{1}{16}$ in., were used for each kind of rivet steel.

The dimensions and details of the butt joints tested in static tension are given in Fig. 2 and those of the butt joints tested in fatigue are given in Fig. 3. Series A and B were driven at the same time as the sample specimens of Group I, whereas series C and D were driven at the same time as the sample specimens of Group II. The main plates of the different series were of ASTM A 7 steel[4] and came from different heats and rollings. The plate surfaces of series A and B had mill scale, were rough, and had a small amount of rust, whereas the plate surfaces of series C and D had mill scale, were smooth, and had no rust. A summary is given in Table I of the physical properties of the main plate materials for the butt joints of series B, C, and D. The joints of series B, C, and D were tested in fatigue and in static tension, whereas those of series A were tested in static tension only.



Series B

Series D



Series C

Series		Areas, sq in.			Type of Rivet Steel	Length of Grip, in.
		Tension	Shear	Bearing		
B	1.00:0.75:1.51	2.67	3.54	1.69	Carbon Alloy	1 5/16 1 5/16
C	1.00:0.75:1.51	2.67	3.54	1.69	Carbon Alloy	4 1/16 4 1/16
D	1.00:1.26:1.83	4.47	3.54	2.44	Carbon Alloy Alloy	1 13/16 1 13/16 2 15/16

FIG. 3.—Dimensions and Details for Specimens of Series B, C, and D.

Rivets—¾ in. in diameter, hot driven with pneumatic hand hammer
Holes—13/16 in. in diameter; series B punched, series C and D drilled
Plate material—ASTM A 7

TABLE I.—PHYSICAL PROPERTIES OF THE MAIN PLATE MATERIALS.

Series	Yield Point, psi	Tensile Strength, psi	Elongation, per cent		Reduction in Area, per cent	Rockwell Hardness[a]
			In 2 in.	In 8 in.		
B......	33 000	63 000	50.0	25.5	56.0	70.0
C......	36 000	64 200	49.5	26.4	55.6	...
D......	31 900	63 200	56.2	32.0	56.2	...

[a] "B" scale, 1/16-in. steel ball, 100-kg load.

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[4] Tentative Specification for Steel for Bridges and Buildings (A 7 - 52 T), 1952 Book of ASTM Standards, Part 1, p. 356.

CLAMPING AND PHYSICAL CHARACTERISTICS OF THE RIVET MATERIALS

The clamping characteristics of both kinds of rivet steels were obtained from the driven rivets of the sample specimens. The clamping stresses for the various lengths of grip were determined by means of relaxation tests. Figure 4 shows the relationship between the clamping stress and length of grip for the alloy steel rivets and the carbon steel rivets of both groups. The clamping stresses of both kinds of rivets increased with an increase in length of grip. At short lengths of grip, the average clamping stress of the alloy steel rivets was about the same as that of the carbon steel rivets. However, at a 4-in. grip, the average clamping stress of the alloy steel rivets was considerably greater than that of the carbon steel rivets. Additional information, as reported by other investigators, concerning the magnitudes of clamping stresses for various types of rivet steels is summarized in Table II.

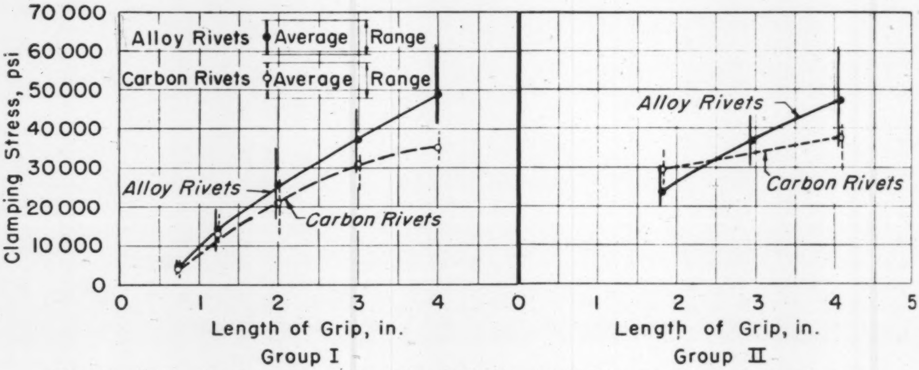


FIG. 4.—Clamping Stress *versus* Length of Grip for Both Groups of Sample Specimens.
All rivets ¾-in. diameter.

No difficulties were encountered in driving the alloy steel rivets or the carbon steel rivets. The time required for driving the various rivets is summarized in Table III. In these tests the alloy rivets were driven as quickly and easily as the ordinary carbon rivets. Additional data concerning the time required for driving various types of rivet steels are reported in the Progress Report of the ASCE Committee of the Structural Division on Structural Alloys (1). A comparison of cost for driving various rivet steels is given in the report and is shown here as Table IV. In the Progress Report it was stated: ". . . if another type of rivet steel could be developed, which would have the strength of the silico-manganese type herein tested, and much greater ease in driving (say only 5 per cent to 10 per cent more costly to drive than carbon steel), such a rivet would have an important economic application, and its cost per pound would not enter materially into the question of its economy."

Tensile properties were determined of the driven and undriven rivets of carbon steel and alloy steel. The tensile properties of the undriven rivet materials were obtained from standard round specimens having a 2-in. gage length. The latter specimens were prepared from undriven rivets chosen at random from the same batches as those used in fabricating the sample specimens and butt joints. The driven rivets were obtained from the sample specimens and were the same as those used for determining the clamping characteristics of the rivets. The tensile properties of the driven and undriven rivets are shown in Table V. The alloy rivet steel met the strength requirements of the ASTM Specification A 195 – 52 T.[2]

TABLE II.—REPORTED VALUES OF CLAMPING STRESS *VERSUS* LENGTH OF GRIP FOR VARIOUS TYPES OF RIVET STEELS.

Sources of Information	Rivet Steel	Method of Driving	Diameter of Rivet, in.	Length of Grip, in.	Average Clamping Stress, psi
W. M. Wilson (2), Univ. of Illinois Eng. Exp. Sta., *Bulletin No. 302*	Carbon	Pneumatic hand hammer	1	3	28 930
				5	33 930
		Hydraulic riveting machine	1	3	31 900
				5	35 980
	Manganese	Pneumatic hand hammer	1	3	23 700
				5	37 280
		Hydraulic riveting machine	1	3	20 030
				5	38 730
W. M. Wilson (3), Univ. of Illinois Eng. Exp. Sta., *Bulletin No. 337*	Low-Alloy Steel A	Pneumatic hand hammer	1	2	5 070
				3	24 600
				5	41 240
		Hydraulic riveting machine	1	2	12 100
				3	20 100
				5	32 380
	Low-Alloy Steel B	Pneumatic hand hammer	1	2	6 300
				3	24 770
				5	34 620
		Hydraulic riveting machine	1	2	17 210
				3	21 770
				5	20 240
	Low-Alloy Steel C	Pneumatic hand hammer	1	2	1 500
				3	14 370
				5	42 370
		Hydraulic riveting machine	1	2	7 510
				3	14 670
				5	30 080
K. H. Lenzen (4), A.R.E.A. *Bulletin No. 480*, June-July, 1949	Carbon (hot-driven)	Hydraulic riveting machine	¾	1½	12 000
	Carbon (cold-driven)	Hanna yoke machine	¾	1½	3 400
C. W. Muhlenbruch (5), *Iron and Steel Engineer*, October, 1949	Carbon (hot-driven)		⅞	6¾	31 050
	Carbon (cold-driven)		⅞	6¾	4 440
	ASTM A 195		⅞	6¾	40 000

For the carbon steel material, the ultimate strength based on the nominal diameter of a rivet was greater after driving by approximately 14 per cent but, based on the actual diameter, little change occurred. In the case of the alloy steel, the ultimate strength based on the nominal diameter was greater after driving by about 22 per cent but based on actual diameters was greater by about 8 per cent. Since the driven rivets had no defined yield point, it was not possible to obtain values of yield stress. Based on changes in length and area, the ductility of the alloy steel rivets was about the same as that of the carbon steel rivets.

Shear strengths were determined for several driven and undriven rivets of both kinds of rivet steel. The strengths were determined for the rivets in double shear by means of a special shear tool and are given in Table VI. The shear strengths of the alloy steel rivets were considerably greater than those of the carbon steel rivets. For both kinds of rivet steels, the shear strengths based on

TABLE III.—TIME REQUIRED FOR DRIVING THE ALLOY AND THE CARBON STEEL RIVETS.

Type of Specimen	Group or Series	Diameter of Rivet, in.	Length of Grip	Average Driving Time per Rivet, sec	
				Carbon Rivet	Alloy Rivet
Sample	I	$\frac{3}{4}$	$\frac{3}{4}$	11 (4)[a]	12 (8)
			$1\frac{1}{4}$	12 (8)	13 (26)
			2	13 (8)	13 (8)
			3	15 (8)	16 (8)
			4	17 (4)	16 (4)
	II	$\frac{3}{4}$	$1\frac{13}{16}$	11 (8)	9 (7)
			$2\frac{15}{16}$	...	12 (5)
			$4\frac{1}{16}$	14 (8)	12 (8)
Butt joints	A	$\frac{3}{4}$	$1\frac{13}{16}$	...	10 (32)
		$\frac{7}{8}$	$1\frac{15}{16}$	...	13 (18)
		1	$2\frac{3}{16}$	...	16 (18)
	B	$\frac{3}{4}$	$1\frac{5}{16}$	10 (26)	10 (27)
	C	$\frac{3}{4}$	$4\frac{1}{16}$	14 (28)	14 (27)
	D	$\frac{3}{4}$	$1\frac{13}{16}$	11 (26)	10 (56)
			$2\frac{15}{16}$	...	11 (28)

[a] Total number of rivets indicated in parentheses.

TABLE IV.—COMPARISON OF COST FOR DRIVING VARIOUS RIVET STEELS.

Kind of Rivets	Rivets Driven in 8 hr	Per Cent Cost
Carbon	1950	100
Alloy, *L*[a]	1550	126
Alloy, *H*[a]	1430	137

[a] *L* and *H* indicate the low and high side of the range in tensile strength considered in ASTM Specification A 195 - 36 T.

TABLE V.—AVERAGE TENSILE PROPERTIES OF THE DRIVEN AND UNDRIVEN RIVETS.

Condition of Rivets	Series	Type of Rivet Steel	Yield Point, psi	Ultimate Tensile Stress, psi		Elongation in 2 in., per cent	Reduction in Area, per cent	Rockwell[a] Hardness
				Actual	Nominal			
Undriven	A	Alloy	60 200	77 400		33.5	63.2	82
	B	Carbon	39 500	65 600		30.1	63.3	61
		Alloy	60 200	77 400		33.5	63.2	82
	C	Carbon	37 700	53 400		44.2	71.2	55
		Alloy	65 600	79 300		34.7	69.3	80
	D	Carbon	48 200	52 700		44.5[b]	74.4	50
		Alloy	67 600	79 200		35.2	69.8	84
Driven	A	Alloy		83 700	94 500	36.0	69.1	89
	B	Carbon		65 200	72 200	33.8	66.8	80
		Alloy		83 700	94 500	36.0	69.1	89
	C	Carbon		53 100	58 400	39.0	73.0	80
		Alloy		82 000	92 500	36.0	70.0	89
	D	Carbon		54 800	64 700	39.0[c]	74.0	64
		Alloy		86 000	98 800	35.0	69.0	90

[a] "B" Scale, $\frac{1}{16}$-in. steel ball, 100-kg load.
[b] 1-in. gage length.
[c] $1\frac{11}{16}$ in. gage length.

actual diameters were slightly changed as a result of the driving operations. The shear strengths based on nominal diameters were appreciably greater than those based on actual diameters.

Hardness surveys were made of the driven and undriven rivets of both kinds of rivet steel, the average hardnesses being given in Table V. For each steel, the distribution of hardness on the midplane of a sectioned rivet was approximately uniform for the driven and undriven rivets. The degree of hole filling for the driven rivets was inspected for several of the fatigue specimens after the fatigue tests were completed. The specimens were prepared for visual inspection by sawing sections containing the longitudinal axes of rivets. These sections were ground and polished and all burrs were removed. The degree of hole filling was about the same for both kinds of rivet steels.

Impact strengths of the driven and undriven rivet materials were determined at room temperature by means of Charpy impact tests of standard V-notch specimens. The impact strengths for the driven rivets of alloy steel were slightly greater than those for the driven rivets of carbon steel. Charpy impact tests of keyhole notch specimens were also made to determine the transition temperatures. The latter tests indicated the following transition temperatures:

Plain carbon steel, after driving	− 57 F
Alloy steel, before driving	−170 F
Alloy steel, after driving	− 90 F

In each case, the transition temperature was obtained from a plot of energy load *versus* temperature and was defined to be the temperature at which 20 ft-lb of energy was required to rupture a specimen. The impact tests showed that the alloy rivet steel had a high notch resistance before and after driving.

A chemical analysis was made of the undriven rivet materials (Table VII).

TABLE VI.—AVERAGE SHEAR STRENGTHS OF THE DRIVEN AND UNDRIVEN RIVETS.

Series	Type of Rivet	Average Shear Strength, Double Shear, psi		
		Undriven Actual Diam	Driven	
			Actual Diam	Nominal Diam
A....	Alloy	56 100	...	...
B....	Carbon	44 600	...	...
	Alloy	56 100	...	...
C....	Carbon	43 300	40 500	44 700
	Alloy	59 800	57 500	66 500
D....	Carbon	39 700	43 600	50 900
	Alloy	59 900	58 600	67 400

[a] $\frac{3}{4}$-in. diameter rivet.

TABLE VII.—CHEMICAL COMPOSITION OF THE UNDRIVEN RIVET MATERIALS.

Type of Rivet Steel	Composition, per cent										
	C	Mn	P	S	Si	Ni	Cr	Mo	Cu	V	Al
Carbon..............	0.14	0.44	0.012	0.030	0.01	0.05	0.04	0.01	0.18	0.005	....
Alloy...............	0.06	0.50	0.020	0.016	1.09	1.29	0.07	0.04	0.68	0.005	0.13

STATIC TENSION TESTS OF JOINTS

The joints of series A were tested in static tension to determine the ultimate strengths, load-slip characteristics, and shear stresses at first major slips of joints fastened with the alloy steel rivets. Three rivet diameters and four rivet patterns were considered for this series of joints. Since a high strength was obtained for the alloy steel rivets, a ratio of tension to shear to bearing of about 1.00 to 1.25 to 1.80 was selected for the joints of series A. Specimens, duplicates of those tested in fatigue, were also tested in static tension. The latter series of tests were made to relate the results of the static tension tests to those of the fatigue tests.

The static tension tests were conducted in a Southwark-Emery testing machine having a 1,000,000-lb capacity. The load on a joint was applied in equal increments until failure occurred either in the main plate of the joint or in the rivets. If failure first occurred in the rivets, the broken rivets were removed and in several cases were replaced with high-strength bolts. The joint was then reloaded until failure occurred in the main plate. Measurements were made of the slippage between the center and side plates of a joint by means of Federal dial gages reading to 0.0001 in. Two gages, one on each edge of a joint, were located at the mid-section of the joint.

In Table VIII are given the ultimate load, the location of failure, and the type of fracture for each joint. In general, the joints of series A with $\frac{3}{4}$-in. diameter rivets failed in the main plates. Each of the latter specimens had one sheared edge and one rolled edge except specimens A4R6 which had rolled edges. The specimens with $\frac{7}{8}$- and 1-in. diameter rivets had machine flame-cut edges and failed by shearing in the rivets. The results for the joints of series A which failed in the main plates are summarized in Fig. 5. The average ultimate stresses on the net sections of these joints are plotted *versus* the ratios of net area to gross area and of edge distance to diameter of rivet. For these joints, the average ultimate stresses decreased with an increase in the above ratios. The ratios of tension to shear to bearing of these joints were about the same. For the specimens which failed in the rivets, the minimum shear-

7

TABLE VIII.—RESULTS OF STATIC TENSION TESTS FOR THE BUTT JOINTS.

Specimen Number	Rivet Diameter, in.	Type of Rivet	Ultimate Load, psi	Stresses at Ultimate Load, psi		Efficiency, per cent	Location of Initial Failure	Type of Fracture, per cent		Edge Condition[c]
				Tension[a] (Net Area)	Shear[b] (Nom. Area)			Cleavage	Shear	
SERIES A—1.00:1.25 TENSION:SHEAR RATIO										
A1L6-1	¾	Alloy	68 600	61 400			Main Plate	50	50	A
2	¾	Alloy	66 500		75 300		Rivets			A
3	¾	Alloy	70 300		79 600		Rivets			A
Average			68 500		77 400					
A2L6-1	¾	Alloy	130 000	58 800			Main Plate	0	100	A
2	¾	Alloy	127 200	57 600			Main Plate	0	100	A
3	¾	Alloy	127 500	57 700			Main Plate	20	80	A
Average			128 200	58 000						
A4L6-1	¾	Alloy	237 000	53 000			Main Plate	0	100	A
2	¾	Alloy	240 000	53 700			Main Plate	0	100	A
3	¾	Alloy	245 000	54 800			Main Plate	0	100	A
Average			241 000	53 900						
A4R6-1	¾	Alloy	243 000	54 400			Main Plate	60	40	B
2	¾	Alloy	248 000	55 400			Main Plate	5	95	B
3	¾	Alloy	246 000	55 000			Main Plate	75	25	B
Average			246 000	55 000						
A2L7-1	⅞	Alloy	173 100		72 200		Rivets			C
2	⅞	Alloy	176 200		73 400		Rivets			C
3	⅞	Alloy	172 800		72 000		Rivets			C
Average			174 000		72 500					
A4R7-1	⅞	Alloy	338 000		70 400		Rivets			C
2	⅞	Alloy	353 500		73 600		Rivets			C
3	⅞	Alloy	349 000		72 700		Rivets			C
Average			347 000		72 300					
A2L8-1	1	Alloy	208 000		66 700		Rivets			C
2	1	Alloy	225 000		72 200		Rivets			C
3	1	Alloy	209 000		67 000		Rivets			C
Average			214 000		68 600					
A4R8-1	1	Alloy	433 000		69 500		Rivets			C
2	1	Alloy	422 000		67 600		Rivets			C
3	1	Alloy	437 500		70 000		Rivets			C
Average			431 000		69 200					
SERIES B—1.00:0.75 TENSION:SHEAR RATIO										
B4R1-C	¾	Carbon	160 000	60 000		70.8	Main Plate	0	100	C
B4R1-A	¾	Alloy	161 000	60 300		71.3	Main Plate	0	100	C
Average				60 200		71.0				
SERIES C—1.00:0.75 TENSION:SHEAR RATIO										
C4R4-C	¾	Carbon	194 500	72 800	45 600	83.0	Rivets	0	100	C
C4R4-A	¾	Alloy	193 000	72 300		82.4	Main Plate	0	100	C
Average				72 600		82.7				
SERIES D—1.00:1.26 TENSION:SHEAR RATIO										
D4R2-C	¾	Carbon	317 000	70 800	50 100	85.5	Rivets	20	80	C
D4R2-A	¾	Alloy	335 000	74 900	76 500	90.5	Rivets	40	60	C
D4R2-B	¾	Alloy	317 000	70 800	74 100	85.5	Rivets	20	80	C
D4R3-A	¾	Alloy	326 000	72 800	70 700	88.0	Rivets	30	70	C
Average				72 300	73 800	87.4				

[a] Failure in main plate.
[b] Failure in double-shear of rivets. Shear stresses based on nominal diameters of rivets.
[c] A = one edge sheared and one edge rolled. B = both edges rolled. C = both edges machine flame cut.

ing strength of the alloy steel rivets was 66,700 psi. This is the same order of magnitude as that reported for other rivet steels meeting ASTM Specification A 195.[2]

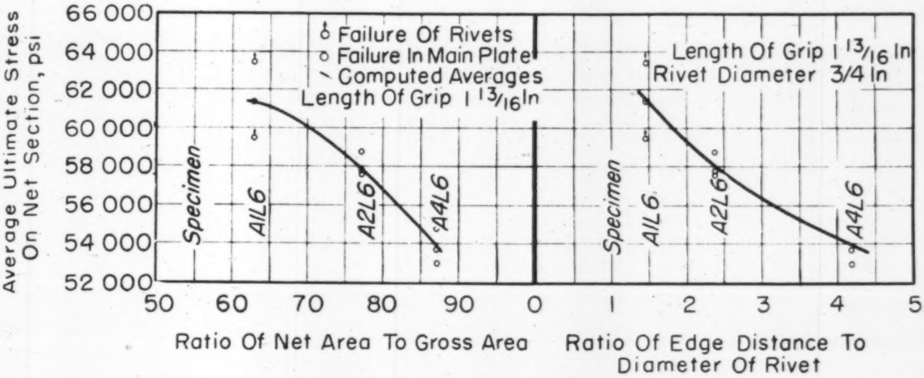


FIG. 5.—Summary of Static Tension Tests for Joints of Series A.

Tension:shear:bearing ratios ~ 100:1.25:1.80.

Efficiencies were obtained for the joints of series B, C, and D. In Fig. 6, the experimental efficiency of each of these joints is plotted *versus* the theoretical efficiency. For the joints having drilled holes, an increase in the theoretical efficiency was accompanied by an increase in the experimental efficiency. The experimental efficiencies of the joints with drilled holes were greater than 82 per cent. The experimental efficiencies of the series of joints having punched holes were appreciably less than those of the series having drilled holes.

In Fig. 7 are shown curves which relate average shear stresses to measured slips for the various joints. The plotted values of slip are the averages of the slips indicated by two dial gages. For ease of comparison, separate groups of curves are drawn for the various types of joints. The behavior of each joint can be described for four ranges of loading as suggested by Davis, Woodruff, and Davis (6). In range 1, the slippage was small and was principally due to the differences in strain of the side plates and the main plate of a joint. At this stage the load was resisted by friction between the plates. In range 2, the friction was overcome and the plates slipped until the rivets came into bearing. At this stage a major slip occurred and was accompanied by an audible "pop." As the load was increased, successive poppings were heard until failure occurred. In range 3, the rivets and plates seemed to deform elastically, and the load-slip relationship was approximately linear. In range 4, yielding of the rivets and plates occurred until the joint failed.

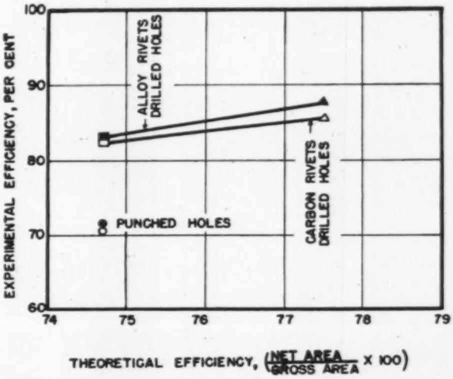


FIG. 6.—Efficiencies for Joints of Series B, C, and D.

A summary is given in Fig. 8 of the shear stresses at which the first major slips occurred for the various joints. (The specimen designation used in Fig. 8 is given in Figs. 2 and 3.) The designation indicates the series, the rivet pattern, and the size and type of rivet. The values of the shear stresses at which the first major slips occurred were greater for the joints of series A having four $\frac{3}{4}$-in. rivets in a line than for the joints of series A having a single $\frac{3}{4}$-in. rivet. The shear stresses at first major slip were less for the joints with the alloy steel rivets having a $\frac{7}{8}$- or 1-in. diameter than for the joints with alloy steel rivets having a $\frac{3}{4}$-in. diameter.

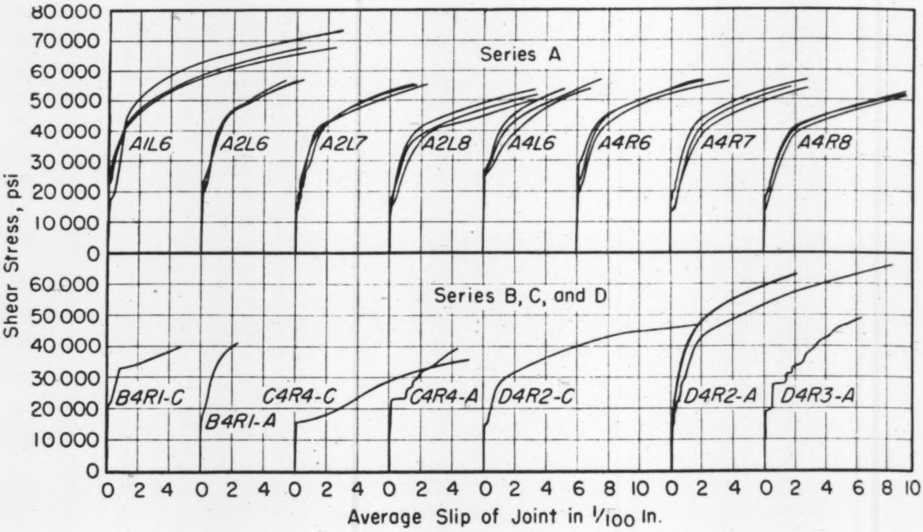


FIG. 7.—Shear Stress *versus* Slip for the Various Butt Joints.

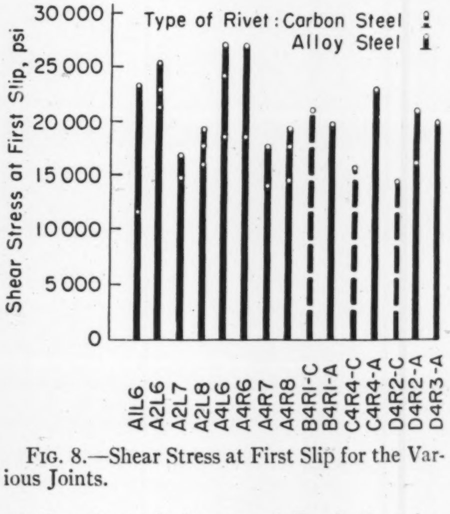


FIG. 8.—Shear Stress at First Slip for the Various Joints.

Apparent coefficients of friction were determined for the joints with $\frac{3}{4}$-in. rivets and are shown in Fig. 9. The coefficients were computed as ratios of the shear stresses at first major slips to the average clamping stresses of the rivets. The coefficients for the joints with $\frac{7}{8}$- or 1-in. rivets could not be determined as no tests had been made to obtain the clamping stresses for these sizes of rivets. The average coefficient of friction for the joints of series A and B was 1.02. This high coefficient may have been the result of the rivets being partially in bearing and of the plate surfaces being rough and slightly rusted. The average coefficient for the joints of series C and D was 0.56.

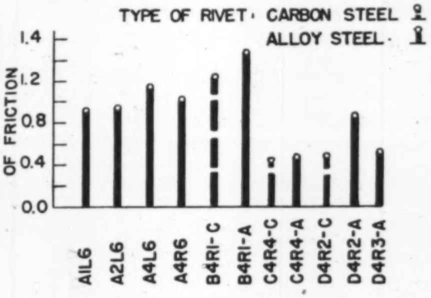
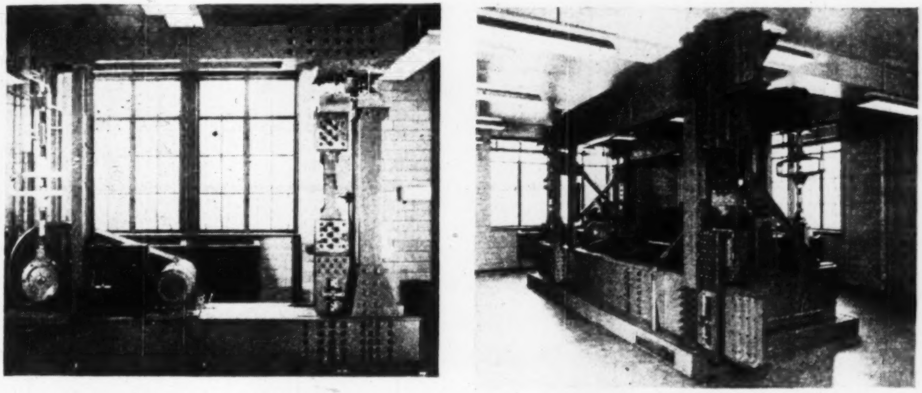


Fig. 9.—Coefficients of Friction for Joints with $\frac{3}{4}$-in. Rivets.


(*a*) 80,000-lb capacity fatigue machine.

(*b*) 250,000-lb capacity fatigue machine.

Fig. 10.—Machines Used for Fatigue Tests.

Fatigue Tests of Series B, C, and D

Several ranges of zero to tension cycles of loading were considered for the butt joints of series B, C, and D. Each joint of these series was fastened with four $\frac{3}{4}$-in. diameter rivets arranged in a rectangular pattern. For each series, both kinds of rivet steel were considered. Series B and C were intended to determine the effect of the kind of rivet steel and the length of grip on the fatigue strength of joints having a ratio of tension to shear of about 1.00 to 0.75. Series D had a ratio of tension to shear of 1.00 to 1.26 and was intended to explore whether a change in the ratio of tension to shear would affect the fatigue strength of joints fastened with the alloy steel rivets.

The fatigue tests were conducted in the 80,000-lb and 250,000-lb capacity machines shown in Fig. 10. The frequency of loading was about 200 cycles per minute for each fatigue machine. The load on a specimen was indicated by a dynamometer which had been calibrated for static loads by means of a standard rectangular specimen. A check was made of the calibration while the machines were in operation. Limit switches were provided to shut off the machines when the elongations of the specimens exceeded a certain amount. Measurements were made of the cyclical slippages occurring between the main plate and side plates of each joint. One gage was mounted on each edge of a specimen and was located at a section midway between the two rows of fasteners.

The results of the fatigue tests are summarized in Fig. 11. The fatigue strengths of the riveted joints increased with an increase in clamping force and with an increase in grip. At a $1\frac{5}{16}$-in. grip, the fatigue strengths of the joints fastened with the alloy steel rivets were about the same as those of similar joints fastened with the carbon steel rivets. However, at grips of $1\frac{13}{16}$ and $4\frac{1}{16}$ in., the fatigue strengths of the joints fastened with the alloy steel rivets were greater than those of similar joints fastened with the carbon steel rivets. An increase in the clamping force resulted in an increase in the fatigue strengths of the joints. A decrease in the ratio of tension to shear of joints fastened with the alloy steel rivets can apparently result in a decrease in the fatigue strengths of the joints.

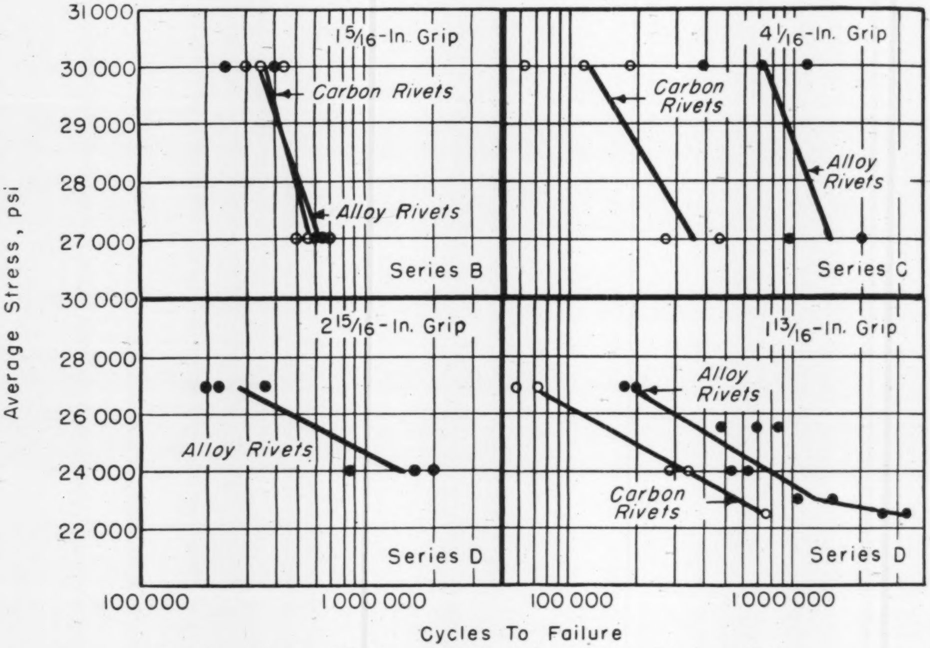


FIG. 11.—Results of Fatigue Tests for Joints of Series B, C, and D.

In general, fatigue fractures of the joints occurred at the net sections of the main plates. Four joints of series D failed in fatigue of the rivets. The latter joints had a $1\frac{13}{16}$-in. grip, a ratio of tension to shear of 1.00 to 1.26, and were tested at a 0 to +27,000 psi range of stress. Two of the four joints which failed in fatigue of the rivets were fastened with carbon steel rivets, and the other two joints were fastened with the alloy steel rivets. The cyclical slippages of all joints were negligible and did not exceed 0.005 in.

CONCLUSIONS

1. The alloy rivet steel reported herein meets the strength requirements of the ASTM Specification A 195 and can be driven as easily and quickly as ordinary ASTM Specification A 141 - 52 T[5] carbon steel rivets.

2. The clamping stress of the alloy steel rivets having a $\frac{3}{4}$-in. diameter increases with an increase in grip and is greater than that reported for other rivets meeting the ASTM Specification A 195. At lengths of grip such as $1\frac{1}{2}$ in., the average clamping stress of the alloy steel rivets having a $\frac{3}{4}$-in. diameter is

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[5] Tentative Specification for Structural Rivet Steel (A 141 - 52 T), 1952 Book of ASTM Standards, Part 1, p. 562.

about the same as that of carbon steel rivets. However, at a 4-in. grip, the average clamping stress of the alloy steel rivets is considerably greater than that of carbon steel rivets.

3. The degree of hole filling and the ductility of the alloy steel rivets are about the same as those of carbon steel rivets.

4. The minimum shearing strength of butt joints fastened with the alloy rivets is of the same order as that reported for other rivet steels meeting the requirements of ASTM Specification A 195. The minimum shearing strength of the joints fastened with the alloy rivets was 66,700 psi.

5 The fatigue strengths of joints fastened with hot-driven rivets increase with an increase in clamping force and with an increase in grip.

6. A decrease in the ratio of tension to shear of joints fastened with hot-driven rivets can result in a decrease in the fatigue strengths of such joints.

Acknowledgments:

Kenneth E. Lenzen, former Research Associate in Civil Engineering at Northwestern University, conducted part of the tests to determine the clamping forces and the tensile properties of the driven rivets. Mr. Lenzen was of great assistance in the initial planning of the program. Acknowledgments are given to the Bethlehem Steel Co. and to its representatives who participated in the program.

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DISCUSSION

MR. SAMUEL EPSTEIN.[1]—Northwestern University had for some time been conducting a study of riveted joints, and when the investigators heard about the new high-strength rivet steel they undertook the study reported in this paper. I think they have done a very excellent piece of work; they have shown that this high-strength rivet, which gives a higher clamping force than the usual high-strength rivets in commercial use, also results in high fatigue strength in the joint.

Metallurgically the higher clamping force is obtained by preventing the formation of a hardened constituent in the rivet underneath the rivet head. The driving temperature of the rivet may be around 2100 F. When the hot rivet is chilled by the cold plate into which it is driven, some hardened constituent may form in the rivet underneath the rivet head, even if the carbon content is not very high. The formation of the hardened constituent is accompanied by an expansion which results in loosening, and hence lowered clamping force. In the new high-strength rivet steel the carbon and manganese contents were kept as low as practicable, the higher strength being obtained through silicon, nickel, and aluminum additions, with the avoidance of manganese and chromium, so as to minimize the chance of the formation of a hardened constituent in the hot-driven rivet.

I should like to ask whether it would be more advantageous to use high-strength bolts instead of high-strength rivets, or in which direction the advantage might lie under different conditions.

MESSRS. FRANK BARON AND EDWARD W. LARSON, JR. (*authors' closure*).—The high-strength bolts which Mr. Epstein mentioned have certain advantages and disadvantages when compared with high-strength rivets. The major advantage of the high-strength bolt arises from the higher clamping forces they can develop as compared with any hot-driven rivets. As a result of the higher clamping forces, bolted joints have a greater fatigue strength than the riveted joints. The higher fatigue strength is partuclarly noticeable at short lengths of grip.

The major disadvantage of the joint fastened with high-strength bolts is the large amount of slippage that can occur once the frictional resistance of the joint is overcome. This large amount of slippage is a result of the clearance between the bolt and the inside of the bolt holes. This clearance is essential for ease of fabrication and would be extremely difficult to reduce. In a riveted joint most of this initial clearance is filled when the rivet is driven.

Thus, the question as to whether it would be more advantageous to use high-strength bolts instead of high-strength rivets may have a different answer depending upon the structure for which the joint is being considered. It is realized that further improvements are needed, especially a rivet which has a high clamping force at short lengths of grip. When such a rivet is obtainable, high-strength riveted joints may prove to be an even more desirable type of connection than they are at present.

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[1] Research Engineer, Bethlehem Steel Co., Bethlehem, Pa.

THE INFLUENCE OF STRAIN RATE AND TEMPERATURE ON THE STRENGTH AND DUCTILITY OF MILD STEEL IN TORSION*

By C. E. Work[1] and T. J. Dolan[1]

Synopsis

An experimental study was made to determine the effect of elevated temperatures and rate of strain on the strength and ductility of SAE 1018 steel in torsion. Cylindrical specimens 0.25 in. in diameter were tested at four different constant strain rates from 0.0001 in. per in. per sec to 12.5 in. per in. per sec and at room temperature, 400, 700, and 1000 F. Two series of tests were conducted: (*a*) Specimens were held at the test temperature for one-half hour before loading, and (*b*) Specimens were given a 200-hr aging treatment at the test temperature before testing.

Torque, angle of twist, and time were continuously recorded and the torsional properties determined. The detailed results are presented in three-dimensional charts and analyzed in terms of mechanisms altering the material behavior. In general, it was found that an increase in strain rate caused an increase in strength, whereas an increase in temperature reduced the strength except in the blue-brittle temperature range. Extremely great ductility was observed at the highest temperature (1000 F), particularly at the slower rates of straining. The 200-hr aging treatment had no appreciable effect on the properties observed.

The experimental observations were compared with several theories that have been proposed to express mathematically the effects of strain rate and temperature on mechanical properties. By proper selection of empirical constants, several equations involving a general relation for flow stress or parameters of a "temperature-modified" strain rate or a "velocity-modified" temperature expressed approximately some of the variations obtained in mechanical properties.

The current needs for operation of structural elements at elevated temperatures accompanied by sudden loading emphasize the importance of determining the influence of strain rate and temperature on structural materials. What constitutes the most appropriate test to determine the properties significant in design? The answer is not obvious since each test has certain disadvantages and limitations.

Published papers describe numerous experiments and propose several theories that contribute a partial understanding of the alterations in properties caused by changing the rate of loading or the temperature. Tension tests have been widely used, and bending tests have been employed frequently in impact studies. However, torsion tests also offer distinct advantages in adaptability to a wide range of strain rates and continuous recording of results. Torsion tests are particularly advantageous in studying inelastic deformations because the length and diameter of the specimens remain nearly constant during twisting to fracture. There is no localization of strain due to necking as in tension testing nor due to a notch as in Charpy bending tests. Thus the strain rate in torsion can be more readily controlled throughout the duration of the test.

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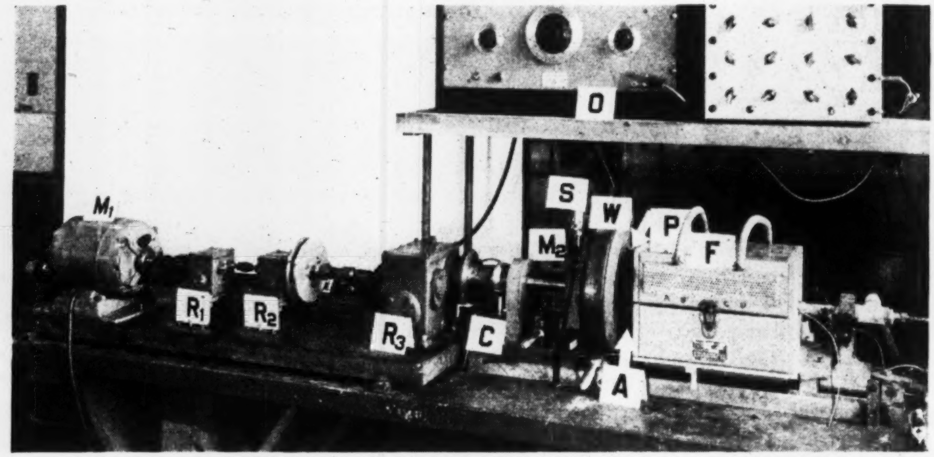
* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Assistant Professor and Research Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Special instruments and equipment were developed at Illinois for torsion tests that made it possible to cover a very broad scope of both variables in the present investigation. Strain rates covered a range of more than five orders of magnitude and ambient temperatures from room temperature up to 1000 F were employed.



M_1, M_2—electric motors
R_1, R_2—speed reducers (50:1)
R_3—speed reducer (67-1/2:1)
F—electric furnace
C—coupling
W—Flywheel
P—Flywheel pins
A—Loading arm
S—Solenoids
O—Audio-oscillator

FIG. 1.—Torsion Testing Machine Set Up for the Slowest Rate of Loading.

SCOPE OF INVESTIGATION

This paper describes the results of an experimental study of the effect of the two parameters, temperature and strain rate, on the mechanical properties of SAE 1018 steel (received in the form of $\frac{5}{8}$ in. hot-rolled bars) subjected to the biaxial state of stress caused by torsion. The chemical analysis of the steel was C 0.16, Mn 0.75, P 0.012, S 0.024, Si 0.04, and the average static tensile properties based on tests of three specimens $\frac{1}{4}$ in. in diameter were as follows:

Yield point	
Upper	38,300 psi
Lower	37,800 psi
Tensile strength	63,500 psi
Elongation (in 1 in.)	36 per cent
Reduction of area	69 per cent

Four rates of torsional strain, differing by factors of 50 to 1, were selected to produce the following nominal rates of shearing strain for the specimens tested: 0.0001, 0.005, 0.25 and 12.5 in. per in. per sec. These are numbered speeds 1, 2, 3, and 4, respectively, in this discussion.

Torsion tests were conducted at room temperature, 400, 700, and 1000 F. For one series, specimens were held at temperature for one-half hour before each test began. A second series was also conducted with specimens given a 200-hr

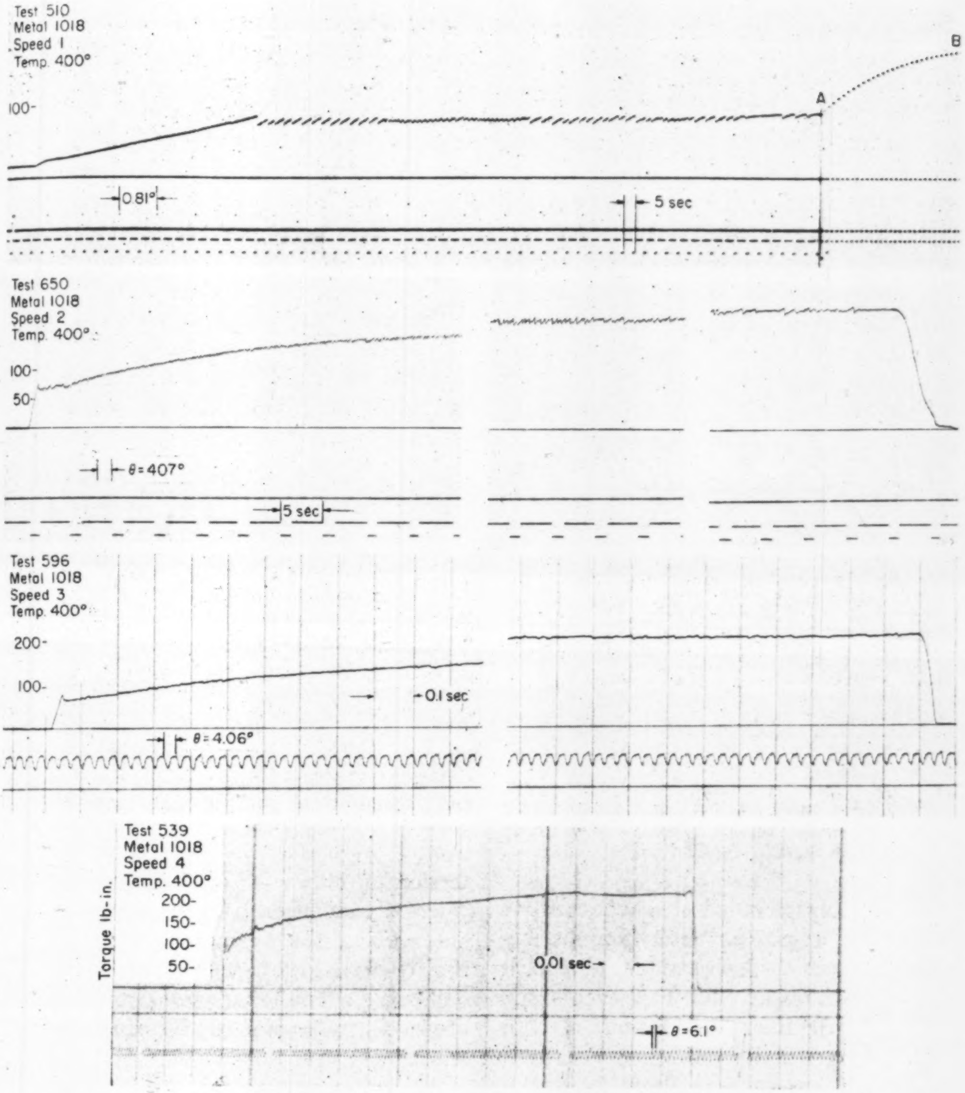


FIG. 2.—Oscillograph Records for Tests of SAE 1018 Steel at 400.
Paper speed changed at *A* in top section; time interval between dots was 1.4 min from *A* to *B*.

aging treatment at the same elevated temperature at which they were subsequently tested. Three specimens were tested for each condition of strain rate and temperature.

The special apparatus and instruments illustrated in Fig. 1 were employed to twist the specimens at the desired rates and temperature and to record continuously measurements of torque, angle of twist, and elapsed time. The specimen, enclosed in the furnace *F* in Fig. 1, is coupled to the flywheel *W* through the loading arm *A* and the pins *P*. The motor M_1 drives the flywheel through a series of speed reducers R_1, R_2, and R_3 for the lowest strain rate. To obtain each higher strain rate, one reducer was removed from the system and the motor moved up to drive the next. For the fastest speed, all reducers were uncoupled and an auxiliary motor M_2 was employed to accelerate the flywheel to the test speed.

The right end of the specimen was gripped in one end of a torque "weighbar." Variable-resistance type wire gages mounted outside the furnace on the surface of the weighbar formed a bridge for measuring the torque. The torque and the output from the various twist measuring instruments employed were continuously recorded on photosensitive paper in a galvanometer-type oscillograph. Figure 2 shows samples of records obtained in tests at 400 F at each strain rate. A detailed description of this equipment will be published elsewhere (**16**)[2] and specific details will not be repeated here.

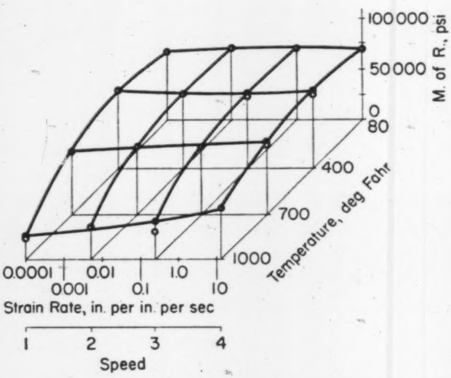


FIG. 3.—Combined Effect of Rate of Strain and Temperature on the Modulus of Rupture of SAE 1018 Steel in Torsion.

The data scaled from each record obtained were plotted in the form of torque-twist curves, and the torsional properties were determined for each specimen. In general, only the average values obtained for each group of three specimens are plotted in the results shown in this paper.

The torsion specimens were reduced to $\frac{1}{4}$ in. in diameter over a length of almost two inches. Large radius fillets at the ends of the reduced section and a gradual relief of 0.003 in. in the central 1-in. test section were necessary to prevent the fracture from occurring at the root of the fillets or at the set screws that were used to attach twist-measuring devices in room-temperature tests.

The yield strength corresponding to 0.2 per cent shear strain offset and the modulus of rupture were arbitrarily computed according to the ordinary torsion formula $\tau = Tc/J$. In instances where a sudden drop in the torque was observed at the beginning of yielding, upper and lower yield points were determined. The lower yield point, when observed, was plotted instead of an offset yield strength.

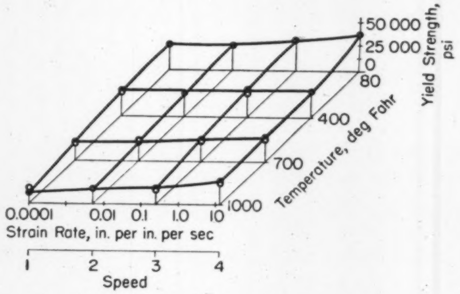


FIG. 4.—Combined Effects of Rate of Strain and Temperature on the Shearing Yield Strength of SAE 1018 Steel in Torsion.

The shearing strain γ was computed from the angle of twist θ in radians within the 1-in. gage length l and for a radius c by the relation: $\gamma = c\theta/l$. Application of this relation for determining strains for large angles of twist may be questioned (**9**). However, it is considered to give an accuracy consistent with other assumptions on which the present calculations are based and is useful here because of its simplicity.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 625.

The values of "energy absorbed" were obtained by integrating graphically, by use of a planimeter, the area under the torque-twist curve. These values represent the *total* energy to fracture for the 1-in. gage length and *not* the energy *per unit volume* as usually reported for tension tests. Because of the radial gradient of strain in the torsion specimens, the energy absorption per unit volume would be highest in the material at the circumference and less for points nearer the axis.

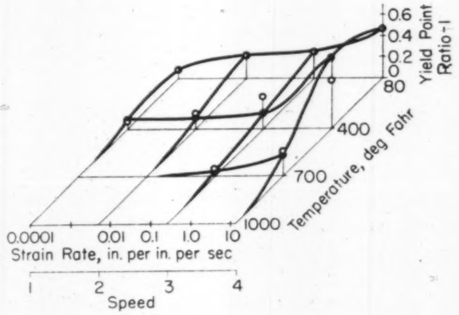

FIG. 5.—Combined Effects of Rate of Strain and Temperature on the Yield Point Ratio for SAE 1018 Steel in Torsion.

EXPERIMENTAL RESULTS

The final results were plotted in the form of three-dimensional graphs and other charts (Figs. 3 to 9) illustrating the variation of torsional properties with the strain rate and temperature.

Strength:

The experimental results plotted in Fig. 3 show that, for tests at room temperature and at 400 F, the modulus of rupture was not affected by pronounced changes in the strain rate. At 700 F there was a decrease of only about 10 per cent in modulus of rupture at the lowest strain rate compared to the highest. However for the slowest tests at 1000 F, the modulus of rupture was less than half that observed at the highest strain rate. Probably 1000 F was sufficiently high to allow a substantial amount of softening or recrystallization; at the slower strain rates, this process had added time to progress more completely.

As may be expected for strain-aging steel, the modulus of rupture was higher at temperatures around 400 F than at any other temperature. The strength was progressively lower for temperatures above 400 F, the minimum strengths being observed at 1000 F at the lowest strain rate.

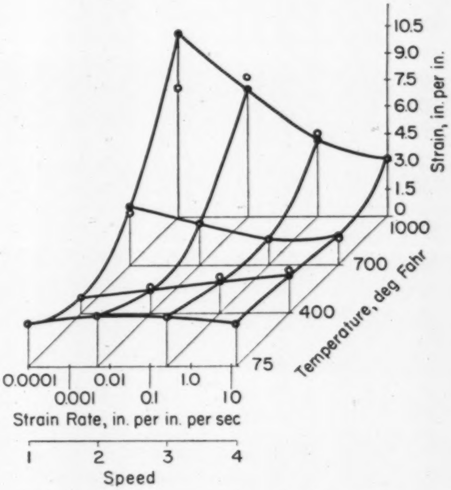

FIG. 6.—Combined Effects of Rate of Strain and Temperature on the Total Shearing Strain of SAE 1018 Steel in Torsion.

The yield strength, Fig. 4 (or particularly the upper yield point), was more sensitive than the modulus of rupture to change in strain rate at the lower temperatures. Initial yielding in this type of steel is through development and propagation of Lüders bands, which is a time-dependent phenomenon. For more rapid loading or lower temperatures, the elastic strengths were increased.

The yield point ratio (defined here as

the ratio of upper to lower value of torque in cases where yielding was accompanied by a sudden drop in the torque) showed considerable sensitivity to both temperature and time (Fig. 5). It is well known that the upper yield point (and hence the ratio) is also affected by factors other than speed and temperature. The yield point is generally considered to be related to strain aging and is associated with the presence of carbon and nitrogen in the steel. The detailed mechanism which results in the observation of a yield point is, however, not clear.

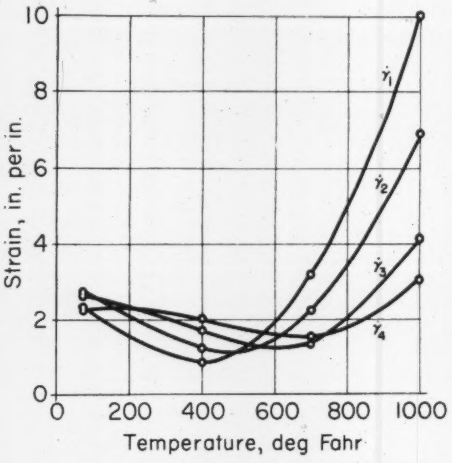

FIG. 7.—Effect of Temperature on the Total Shearing Strain of SAE 1018 Steel in Torsion.

Ductility:

The ductility as measured by the total shearing strain was found in general to be influenced sharply by the blue-brittleness effect at intermediate temperatures. These trends are illustrated in Figs. 6 and 7. A reduction of ductility as a result of blue brittleness is indicated by the valley extending diagonally across the three-dimensional plot in Fig. 6. The hill in the background indicates the sharp increases in ductility observed when temperatures were increased above the blue-brittleness temperature. The slopes in the foreground were less pronounced illustrating less variation in ductility for temperatures below the blue-brittleness range.

Quantitatively, this variation in ductility was from about 2.5 in. per in. shearing strain for all strain rates at room temperature to minima ranging from 0.9 in the slow tests to 1.6 in fast tests at the blue-brittleness temperatures. Maximum values of strain were observed at 1000 F, but ranged from three in the high speed test to about ten at the slowest speed.

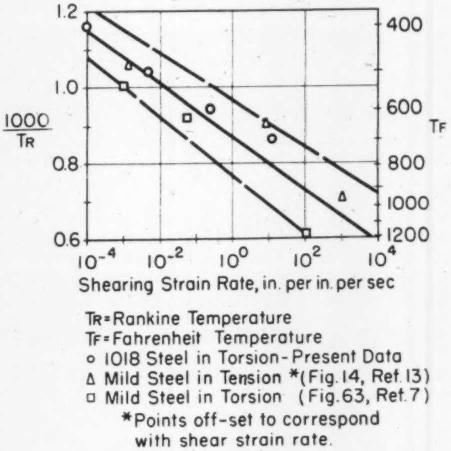

FIG. 8.—Shift of Blue-Brittleness Temperature for Steel with Change of Strain Rate.

The zone of minimum ductility covered a fairly wide band of temperatures for any strain rate; in Fig. 7 the minimum point for one set of data might be chosen anywhere within a range of a hundred or more degrees. An increase in strength frequently accompanies the loss in ductility in this temperature range but was not observed to be pronounced in these experiments.

Blue brittleness is considered to be the result of strain aging (**3**, **8**) and the temperature at which it occurs is a function of the rate of straining. Figure 8 shows the temperature for minimum ductility from the curves in Fig. 7 plotted *versus* rate of strain. For the sake of comparison, a few points obtained from other tension and torsion tests (**7, 13**) are also included in Fig. 8. Data from all three sources fall within the same general scatter band represented by the dashed lines. The general trend of this scatter band can be expressed mathematically in the form

$$A = \dot{\gamma} e^{\frac{B}{T_R}} \ldots\ldots\ldots\ldots (1)$$

$$T_R = \frac{B}{C - \ln \dot{\gamma}} \ldots\ldots\ldots\ldots (2)$$

where:

A, B, and C = constants,
$\dot{\gamma}$ = shearing strain rate, and
T_R = absolute temperature in Rankine units.

Evaluating the constants for the solid line in Fig. 8, we find $A = 1.77 \times 10^{12}$, $B = 3.25 \times 10^4$, $c = \ln A = 28.2$. Equation 2 then becomes

$$T_R = \frac{32{,}500}{28.2 - \ln \dot{\gamma}} \ldots\ldots\ldots\ldots (3)$$

Other investigators (**3**) report qualitative agreement that the blue-brittle temperature increases with rate of strain, but in most cases the actual rates of strain are not reported. The scarcity of strain rate data is probably due to the fact that it is difficult to determine reliably the actual rate of strain in the bending impact test or in tension tests in which necking occurs.

Effect of the Aging Heat Treatment:

There was no reason to expect the 200-hr aging treatment to have a marked effect on this steel. The only possible change might be a quench aging resulting from air cooling after the hot-rolling operation that would be accelerated slightly by the 200-hr heating.

Average values for specimens heated to the test temperature for 200 hr in advance of the actual test are plotted as open circles in Figs. 3, 4, 5, 6, and 9, whereas the data for unaged specimens are plotted as solid circles.

Comparisons of the aged and unaged specimens in Figs. 3, 4, and 6 indicate that if an aging effect were present, it was too small to be distinguished from the normal scatter inherent in the experimental measurements. The apparent differences in ordinates observed in Fig. 6 between aged and unaged specimens tested at 1000 F are probably the result of plotting averages, each based on two or three events which are part of a widely distributed population. For example, when the individual specimens tested at 1000 F were compared, wide scatter was observed. It appeared likely that any apparent difference between the averages for the aged and unaged specimens was probably less than the statistical or chance variations exhibited by the individual specimens. Since large variations in total strain occurred for the slow loading at 1000 F, more than three samples were needed for the mean value to represent a reliable average. The scatter in the total strain measured for individ-

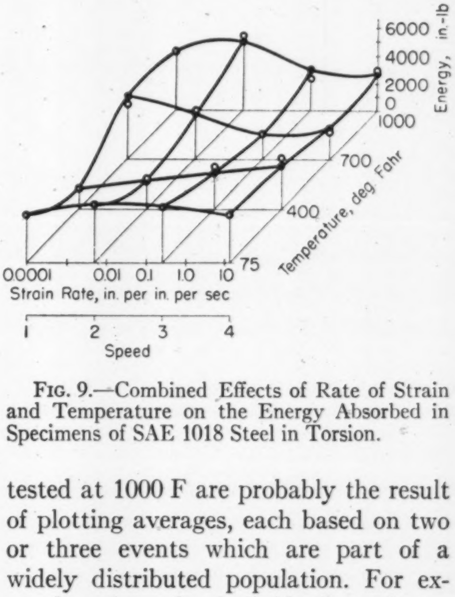

FIG. 9.—Combined Effects of Rate of Strain and Temperature on the Energy Absorbed in Specimens of SAE 1018 Steel in Torsion.

ual specimens at the three lower temperatures was very much less than that observed at 1000 F.

The few effects of aging on the yield point ratio apparent in Fig. 5 also are probably due to chance, since any deviations which occurred were exaggerated by subtraction of two numbers of the same relative magnitude (Y.P. Ratio −1). The shapes of the torque-twist diagrams in the region of initial yielding were not noticeably altered by the 200-hr heat treatment.

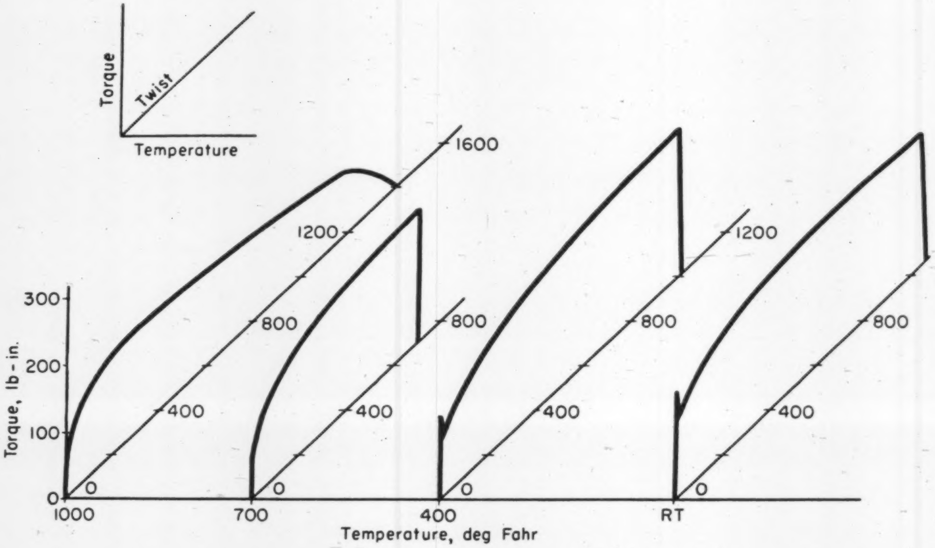

FIG. 10.—Torque-Twist Curves for Fourth Speed Torsion Tests of SAE 1018 Steel at Four Temperatures.

Energy Absorption:

The relative energy absorbed by a specimen under the different testing conditions varied as a combination of the trends observed for the influence of strain rate and temperature on the modulus of rupture and on the maximum strain. This follows directly because the relationship of the energy absorbed to the twisting moment, T, and the corresponding angle of twist, θ, expressed in radians, is:

$$\text{Total energy absorbed, in torsion} = \int_0^{\theta_1} T\, d\theta \ldots\ldots (4)$$

The integral represents the area under the torque-twist diagram up to the twist θ_1. For the shapes of diagrams obtained for the low-carbon steel, the absorbed energy varied in proportion to the product of the maximum torque and the total angle of twist to fracture. Therefore, discussions of relative strength and ductility can be extended to apply to the relative energy absorption for the various testing conditions.

The influence of temperature and strain rate on the energy absorbed in fracturing specimens is illustrated in Fig. 9. Because any change in temperature or strain rate produced opposite effects on ductility and strength (a large increase in ductility was modified by a decrease in strength), no conspicuously large values of absorbed energy were observed.

Shape of the Torque-Twist Curves:

Figure 10 illustrates the effect of elevated temperatures on the shape of the torque-twist curves for the *highest* strain rate.[3]

The mode of fracture progression controlled the shape of the final portion of the torque-twist curves. At the lower temperatures the failure was sudden and the torque dropped to zero almost instantaneously. At higher temperatures and slower speeds the final failure was less rapid and the records showed that the torque gradually tapered off to zero.

Continuous torque recordings at 400 (Fig. 2(top)) and at 700 F exhibited the erratic torque, characteristic of the material behavior in the blue-brittle range. This recurring process of build up and sudden drop of torque would not be so clearly observed in larger specimens where an averaging tendency masks out the individual jumps. Hall (4) watched this process in thin strip specimens and could trace the advance of a Lüders band with the drop in stress followed by a build up as soon as one band was arrested. When the stress reached a sufficiently large magnitude, a new Lüders band darted through the metal, etc. This phenomenon is observed only for a limited range of combinations of temperatures and strain rates; it is undoubtedly related to the rates of strain aging and of slip within the crystal structure. Portions of records from torsion tests are shown for all four strain rates at 400 F in Fig. 2. The appearance in the recording of the irregularities is influenced by the rate of travel of the paper in the camera of the oscillograph as compared with the rate of strain in the specimen. The torque scale, the time intervals, and the twist increments are labeled to aid in interpreting these records.

CORRELATION OF STRAIN RATE AND TEMPERATURE EFFECTS WITH MATHEMATICAL THEORIES

It appears probable that no complete and acceptable theory can be developed to express all the complex effects of strain rate and temperature on the mechanical properties of structural metals. Nevertheless, it was felt desirable to investigate the usefulness of several proposed relationships in expressing mathematically the variations observed in these experiments.

Simplification Used:

Theoretical considerations for correlation of data of this type are usually intended to determine mathematical expressions relating the flow stress to the strain, rate of strain, temperature and certain material constants. The equations developed are generally stated in terms of the "true" stress. However, it was more convenient here to work in terms of applied torque instead of stress. Arbitrary substitution of torque for shearing stress did not seem justified since the two are not equal or even proportional during plastic deformation.

For the plastic range, Eq 21 of the Appendix can be used to evaluate the shearing stress at the surface of a specimen. This equation for the shearing stress τ includes a constant coefficient $K' = \frac{1}{2}\pi c^3$, a term involving the torque T, and a term involving another variable torque T_0.

$$\tau = K'(4T - T_0) \qquad \text{(5)}$$

Computations showed that for a given or constant θ (of the magnitudes examined in the following sections), the amount by which T_0 differed from being proportional to T was small enough compared to $4T$ that valid observations for the plastic range were possible by using the following simplification:[4]

---

[3] At the slower speeds of straining, less drop in torque was observed at the yield point; a somewhat greater decrease in torque occurred after the maximum was reached early in the test, and there were considerable variations in strength and ductility as discussed earlier.

$$\tau\Big|_{\theta} = KT \ldots\ldots\ldots\ldots\ldots (6)$$

In order to substantiate this reasoning, the same types of plots as those shown in Figs. 11 to 14 were constructed for representative sample conditions. The curves resulting showed the same trends and scatter when values of the torque T were plotted as when the more rigorous analysis employing values of the shearing stress τ were used.

Equivalent Strain Rate Parameter:

A quantitative relation between the effects of temperature and strain rate has been suggested by Zener and Hollomon (**18**). The flow stress σ at a given strain ϵ was expressed by the function

$$\sigma\Big|_{\epsilon} = f\left(\dot{\epsilon}e^{\frac{Q}{RT}}\right) = f(P) \ldots\ldots\ldots (7)$$

where:

$\dot{\epsilon}$ = strain rate,

R = universal gas constant, and

Q = constant depending on the material.

For low temperatures Q has been measured (**6**) to be about 10,000 cal per g-mol for several steels, and since R is 1.987 cal per deg-mol, the exponent becomes roughly $5000/T$. In terms of shearing strain rate, the parameter P then has the value

$$P = \dot{\gamma}e^{5000/T} \ldots\ldots\ldots\ldots\ldots (8)$$

Here P is a strain rate modified by a dimensionless coefficient which is a function of temperature, or P is an equivalent strain rate. Using Q as 10,000, this concept was tested with the present data and found to give too little weight to the effect of temperature. For elevated temperatures, however, Q is probably not a constant but depends on stress and temperature as well. By trial and error a magnitude can be selected for Q to cause the data to fall along a fairly smooth curve. Figure 11 shows the torque corresponding to a shearing strain of 0.50 in. per in. plotted to log scale against the parameter P based on Q/R = 40,000.

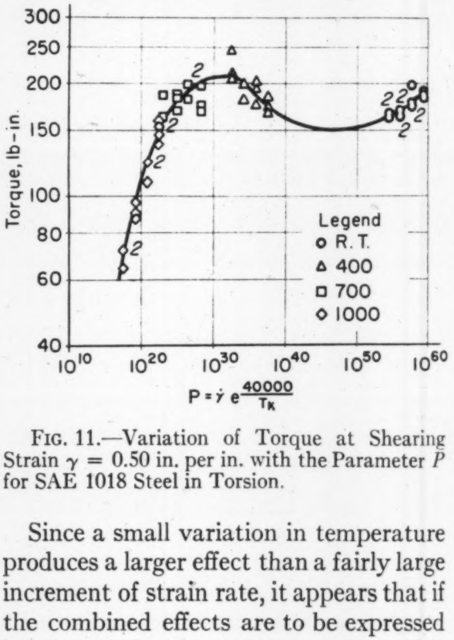

FIG. 11.—Variation of Torque at Shearing Strain γ = 0.50 in. per in. with the Parameter P for SAE 1018 Steel in Torsion.

Since a small variation in temperature produces a larger effect than a fairly large increment of strain rate, it appears that if the combined effects are to be expressed in terms of only one parameter, this parameter should be more closely associated with temperature with an appropriate factor to account for strain-rate effects.

General Equation for Flow:

The general expression for flow stress as a function of strain, strain rate and temperature proposed by Hollomon and Lubahn (**11**) is:

$$\sigma = CG^{T_R}\left(\frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right)^{DT_R} \epsilon^{\left(E - FT_R \ln\frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right)} \ldots . (9)$$

---

[4] In the notation used here, a vertical line is followed by one or two symbols to indicate the variables that are held constant for a given equation. In Eq 6 the shearing stress τ is expressed as a function of the torque T for a constant angle of twist θ. Other variables such as strain rate and temperature are not restricted in this equation.

Correlation of the effects of rate of strain and temperature with the present data can be examined by use of a set of three simplified equations in which different pairs of variables are held constant. For use here, shearing stresses and strains have been substituted for normal stresses and strains and the temperature T_R is expressed according to the Rankine scale. Written in logarithmic form and calling the resulting constants C_1 to C_6 we have

$$\ln \tau \Big|_{\dot{\gamma}, T_R} = C_1 + C_2 \ln \gamma \; \ldots\ldots (10)$$

$$\ln T \Big|_{\gamma, T_R} = C_3 + C_4 \ln \dot{\gamma} \; \ldots\ldots (11)$$

$$\ln T \Big|_{\gamma, \dot{\gamma}} = C_5 + C_6 \, T_R \; \ldots\ldots (12)$$

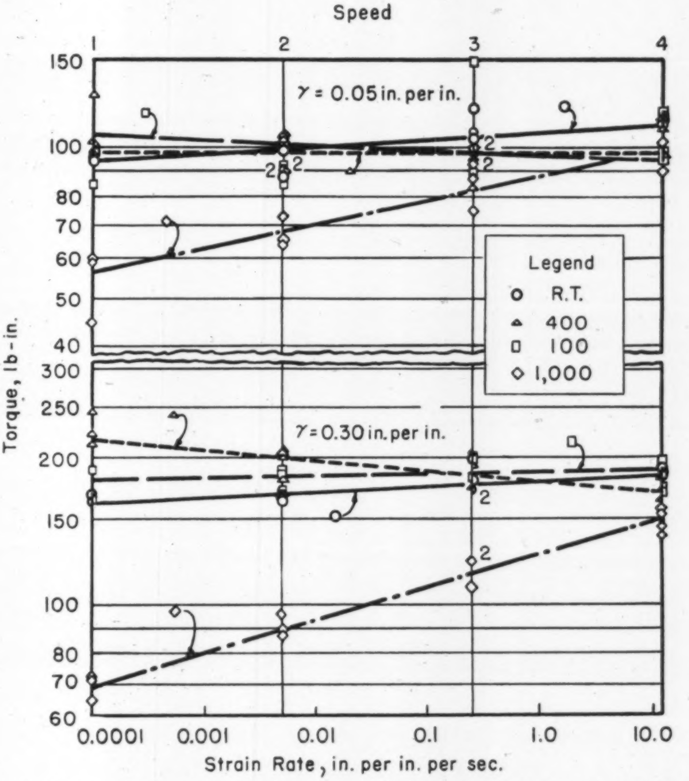

FIG. 12.—Effect of Rate of Strain on the Torque at Constant Shearing Strain in Torsion Tests of SAE 1018 Steel.

The latter two equations are of primary interest here.

Constant Strain and Temperature:

Equation 11 relates the effect of strain rate $\dot{\gamma}$ to the flow stress or torque for a constant shearing strain γ and at constant temperature T_R. This equation indicates a linear relationship between ln T and ln $\dot{\gamma}$. Hence a plot of ln T *versus* ln $\dot{\gamma}$ (for γ and T_R constant) should make the experimental values fall on a straight line for Eq 11 to be satisfied. Since the four speeds employed in the present tests each differed by factors of 50 to 1, the speed numbers can be used to represent the logarithm of the strain rate.

In Fig. 12 the torque is plotted to a log scale *versus* the strain rate. Each

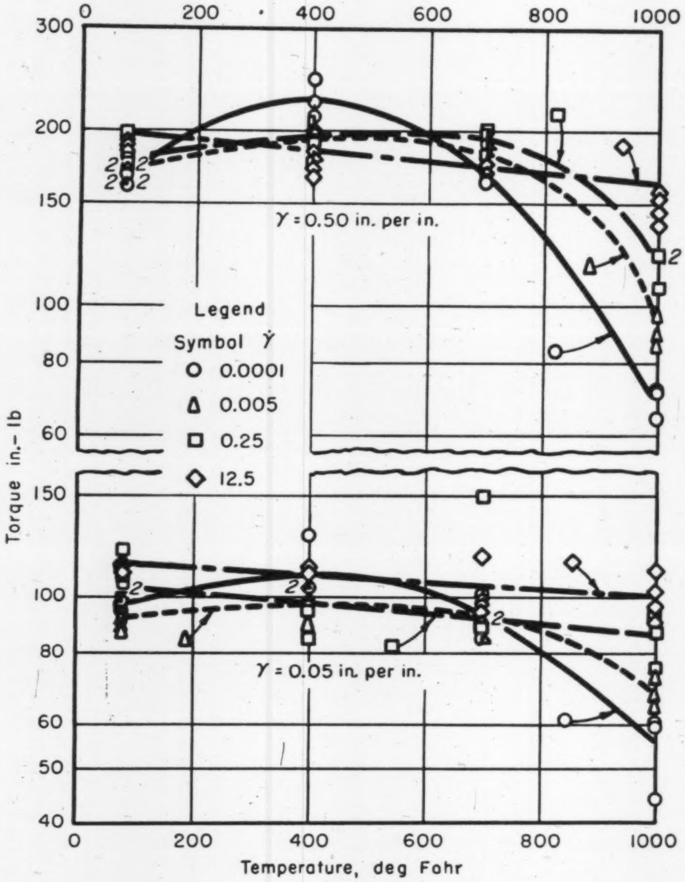

FIG. 13.—Effect of Temperature on the Torque at Constant Shearing Strain in Torsion Tests of SAE 1018 Steel.

point represents the results obtained from one specimen. Data are shown for two values of shearing strain, namely, $\gamma = 0.05$ and 0.30. Several other values of strain were also studied with results similar to those shown.

The curves obtained are nearly straight for all temperatures for the values of γ checked; that is, the variation of the logarithm of stress or torque with the logarithm of strain rate is nearly linear. These data indicate then that although the effect of strain rate is in general not large, the variations in strength of this metal in torsion with changes in strain rate can be predicted fairly well by the relation:

$$T\Big|_{\gamma, T_R} = c_1 \dot{\gamma}^{c_2} \quad \ldots\ldots\ldots\ldots (13)$$

where c_1 and c_2 depend on the strain and temperature.

Constant Strain and Strain Rate:

Equation 12 relates the torque or stress to the temperature for constant strain and strain rate. The logarithm of the torque T is expressed as a linear function of the absolute (Rankine) temperature T_R. This can be checked by plotting the torque to a log scale *versus* T_R or, more conveniently, *versus* the Fahr temperature T_F. (Since $T_R = T_F + 459.6$, the difference between T_F and T_R may be included in the constant c_5 of Eq 12).

Some of the data plotted in this manner are included as Fig. 13. The trends of the points in general appear to form rather definite curves which *cannot* be considered linear. Therefore it must be concluded that Eq 12 is inaccurate in expressing the variations of strength properties caused by temperature changes. The patterns in which test points fall for aged specimens were similar to those shown for the unaged specimens.

Thus the effect of temperature for the ranges of variables studied here did not conform well with the theoretical relation expressed in Eq 12. The reason for this inaccuracy may be attributed to such actions as strain aging or precipitation and coalescence of solute atoms at certain temperatures. In other words, the properties changed somewhat during the progress of the test and could not be expressed by a single simple equation that is independent of the parameter of time.

Velocity-Modified Temperature Parameter:

A "velocity-modified" temperature parameter has been proposed by MacGregor and Fisher (12). Instead of expressing the flow stress as a function of three variables (temperature, strain rate and strain) as:

$$\sigma = f(T_R, \dot{\epsilon}, \epsilon) \ldots\ldots\ldots\ldots (14)$$

they propose that the first two be combined in a velocity-modified temperature, T_m leaving

$$\sigma = f(T_m, \epsilon) \ldots\ldots\ldots\ldots (15)$$

An expression for determining T_m was also proposed:

$$T_m = T_R\left(1 - k \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right) \ldots\ldots (16)$$

where:

$\dot{\epsilon}_0$ = arbitrary reference strain rate and

k = constant for the material selected (presumably by trial and error) so that when stress is plotted *versus* T_m, a smooth curve results. This concept was examined by Mac Gregor and Fisher by comparison with tension data; they reported general agreement for the materials and range of variables covered.

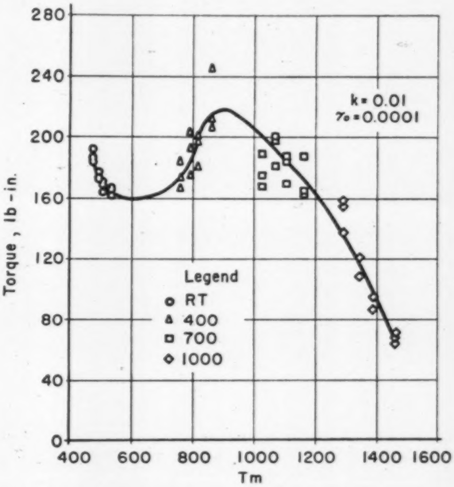

FIG. 14.—Variation of Torque at Shearing Strain, $\gamma = 0.50$ in. per in. with the Parameter T_m for SAE 1018 Steel in Torsion.

For comparison with torsional data the shearing strain rate may be substituted and the Rankine scale for absolute temperature T_R selected. The shearing stress in torsion, according to this concept would be

$$\tau = f(T_m, \gamma) \ldots\ldots\ldots\ldots (17)$$

This concept was examined by using the approximate relationship of torque T to shearing stress τ. Experimental values were plotted to obtain curves for several different values of shearing strain γ illustrating the relationship:

$$T\Big|_{\gamma} = f(T_m) \quad \ldots\ldots\ldots\ldots (18)$$

To determine T_m, the slowest strain rate was selected as the reference so that $\dot{\gamma}_0 = 0.0001$ in. per in. per sec. The value of k in Eq 16 which gave the smoothest curves was 0.010 for this material.

The chart for torque *versus* T_m for one value of strain γ is included in Fig. 14. It can be seen that there is fair general agreement with Eq 18 since the points group around the relatively smooth curve sketched in to follow the average trend. The shape of the curve thus obtained would not be the same for different metals, but no attempt is made by the theory to predict this shape.

Correlation was noticeably poorer when the yield point or the modulus of rupture was plotted instead of the flow stress or torque at constant strain. A very broad band was required to envelop the points thus obtained although the general course followed by the band was similar to that for the curves shown in Fig. 14.

These data indicate that by proper choice of empirical constants an approximation to the effects of strain rate or temperature may be expressed for limited conditions in terms of a single parameter, the so-called velocity-modified temperature. No simple relationship exists, however, to express the relation between this parameter and the flow stress or torque for a given twist.

CONCLUSIONS

For the range of variables covered in this study, the following conclusions seem justified:

1. Increasing the temperature caused a general decrease in strength for all rates of strain.

2. Increasing the rate of strain usually caused an increase in strength.

3. For a range of elevated temperatures beginning at about 400 F, a reduction of ductility (usually referred to as blue brittleness) and a slight increase in strength were observed. Above this range a pronounced increase in ductility was observed with increase in temperature and decrease in strain rate.

4. The absolute (Rankine) blue-brittleness temperature T_R was a function of the rate of shearing strain as expressed roughly by the equation:

$$T_R = \frac{32{,}500}{28.2 - \ln \dot{\gamma}}$$

5. The torque-time records obtained in the blue-brittleness range exhibited jagged or erratic variations in flow stress.

6. The rate of increase of flow stress τ (or torque T) with increase in strain rate $\dot{\gamma}$ at a fixed strain γ and temperature T_R can be expressed by the equation:

$$\tau\Big|_{\gamma, T_R} = c_1 \dot{\gamma}^{c_2}$$

where c_1 and c_2 are empirical constants which depend on the temperature and the amount of strain.

7. The rate of decrease of flow stress τ (or torque T) for a given strain with increase in temperature did not conform with the behavior predicted by the equation:

$$\tau\Big|_{\gamma, \dot{\gamma}} = j e^{h T_R}$$

where j and h are constants which depend on the material, strain, and strain rate.

8. It is possible to combine the effects of strain rate and temperature in a single parameter (the velocity-modified temperature T_m or the equivalent strain rate P) and get a smooth curve for flow stress at a given plastic strain. The shape of the curve obtained (or the variation with strain) was not readily expressible.

9. The ratio of the upper to lower yield point torques was found to de-

crease with an increase in temperature, and decreased or disappeared at the lower strain rates.

10. A 200-hr aging treatment (at the test temperature) in advance of the test had no appreciable effect on the strength or ductility of the steel for any of the temperatures or strain rates studied.

Acknowledgment:

The research described in this paper was conducted in the laboratories of the Department of Theoretical and Applied Mechanics as a portion of the work of the Engineering Experiment Station at the University of Illinois in cooperation with the Wright Air Development Center Ohio (**17**) under contract AF-33 (038)-21587. Special acknowledgment is due to F. C. Rally, T. Dimoff, G. L. Lovestrand, D. C. Simpson, M. Sugi, P. Domotor, and R. D. Field who have helped with the experimental phases of this investigation.

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APPENDIX

EQUATION FOR SHEARING STRESS IN TORSION

Computation of the actual shearing stress at any point in a member subjected to torsion can be made using the well-known $\tau = \frac{Tc}{J}$ relationship only for stresses which remain proportional to the corresponding strains. When this equation is used for torques exceeding the limit of proportionality, the nominal value resulting is not a real stress but merely represents the torque converted to stress units by dividing by the section modulus $\frac{J}{c}$ which is a function of the dimensions of the cross-section.

A relationship was developed to express the true shearing stress τ at the surface of a cylindrical bar of radius c subjected to pure torsion as a function of the twisting moment T, the angle of twist or shearing strain γ, and the rate of increase in moment with respect to twist or strain $\frac{\delta T}{\delta \gamma}$. This relation for torsion is similar to the equation for bending developed by Herbert (5). It is based on the assumptions of homogeneity and isotropy of the material and applied to a bar of uniform circular cross-section at a point sufficiently removed from the point of application of the load so that stress is not a function of axial position.

The relationship derived is usually defined in the form

$$\tau = \frac{1}{2\pi c^3}\left(3T + \gamma \frac{\delta T}{\delta \gamma}\right) \ldots\ldots (19)$$

Equation 19 provides an expression from which one can determine the shearing stress τ in the outer fibers for any γ from test data giving the relation between the twisting moment T and the shearing strain γ in the outer fibers.

By noting that $\frac{\delta T}{\delta \gamma}$ is the slope of the T *versus* γ curve and, hence, for any point on the curve

$$\frac{\delta T}{\delta \gamma} = \frac{T - T_0}{\gamma} \ldots\ldots\ldots\ldots (20)$$

where T_0 is the intercept of the tangent on the T axis, we can substitute this relation in Eq 19 and obtain

$$\tau = \frac{1}{2\pi c^3}(4T - T_0) \ldots\ldots\ldots (21)$$

Equation 21 gives the relationships in the most convenient form for use in determining τ from the T *versus* γ curve.

Since γ was defined to be proportional to θ and since plots of T *versus* θ were made as a part of routine calculations of data, true stresses computed were determined from the T *versus* θ curves available rather than replotting T *versus* γ.

CREEP AND CREEP-RUPTURE OF SOME FERRITIC STEELS CONTAINING 5 PER CENT TO 17 PER CENT CHROMIUM*

By E. J. Dulis[1] and G. V. Smith[1]

Synopsis

Data on creep and creep-rupture properties are presented for eleven steels ranging in alloy content from 5 Cr, 1/2 Mo to 17 Cr, with additions of silicon, titanium, columbium, or boron to some. Microstructural changes, observed after test, including carbide spheroidization and recrystallization, are described. Some observations on the mechanism of flow and fracture of ferritic and austenitic steels are included.

For applications where service conditions are not severe enough to require the austenitic 18 per cent chromium, 8 per cent nickel type steels, chromium-molybdenum or straight chromium steels may be used. Choice of a particular grade is dependent on the service conditions under which the steel is to be used. The 5 Cr, ½ Mo grade is widely used in equipment for service up to 1200 F under corrosive and oxidizing conditions. Additions such as silicon, titanium and columbium impart to this grade, certain properties which are desirable for specific applications; silicon increases oxidation resistance, and titanium and columbium reduce tendency to air hardening, thereby improving weldability.

The 8 Cr, 1 Mo grades have relatively good oxidation resistance up to 1300 F, but their creep strength decreases rapidly above 1100 F. Higher chromium grades, such as 12 Cr or 12 Cr, ½ Mo types, are employed when still better oxidation resistance is needed, but when strength is a factor they are seldom used above 1200 F.

Nonhardenable 17 Cr, Type 430, steel has oxidation resistance similar to 18 Cr, 8 Ni up to 1500 F; however, its low strength at elevated temperatures and susceptibility to so-called 885 F embrittlement limits the use of this steel to applications where strength and toughness are not primary requirements.

Materials and Procedure

The steels used in this investigation, Table I, include both commercial and experimental grades received in the form of 1-in. round or square bars. Steels B, E, F, G, and H were tested in creep and creep-rupture while the rest were tested in creep-rupture only.

Chemical composition, heat treatment, grain size, and initial hardness are given in Table I. Steels A, C, D, and K were heat treated at the National Tube Division, Lorain Works and the rest at this laboratory prior to machining into test specimens. All of the steels were initially fine grained except D (5 Cr, ½ Mo (Ti)) which had a duplex grain size.

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Short-time tensile properties of steels E and F at both room temperature and at elevated temperatures were available from a previous study and are given in Table II.

Creep and creep-rutpure tests were made by methods which have been discussed in detail in an earlier publication (**1**).[2] Creep extension was also measured in some creep-rupture tests by sighting with a microscope through the furnace window upon reference marks on a wire moving within a tube, both of a platinum-10 per cent rhodium alloy, spot-welded to the shoulders of the specimen (**2**). This method was used for the principal purpose of obtaining accurate values of the creep intercept (see subsequent discussion). The creep intercept depends in part upon the strain that occurs during loading, which cannot be accurately determined from the fall in the loading lever because of the strain which takes place throughout the test stand when the load is applied. Although an approximate correction can be made for this strain in the test stand itself, it was deemed desirable to obtain more accurate values of the strain in the specimen by direct measurement. Once the load is applied, further strain is essentially confined to the specimen, and thus creep rates computed from the autographic record are quite accurate and have been found to agree closely with those obtained by direct measure-

TABLE I.—CHEMICAL COMPOSITION, HEAT TREATMENT, AND INITIAL HARDNESS AND GRAIN SIZE OF STEELS TESTED.

Steel	Type	Chemical Composition, per cent										ASTM Grain Size	Diamond Pyramid Hardness	Heat Treatment, deg Fahr
		C	Mn	P	S	Si	Cr	Mo	N	Al	Other	Before Test		
A	5 Cr, ½ Mo	0.13	0.42	0.030	0.017	0.22	4.69	0.47	0.021	0.004		6–8	124	1550 F.C.
B	5 Cr, ½ Mo (Si)	0.13	0.26	0.011	0.010	1.57	5.24	0.50	...	...	...	8–9	168	1600 F.C.
C	5 Cr, ½ Mo (Cb)	0.09	0.54	0.027	0.018	0.41	5.05	0.53	0.012	0.065	0.89 Cb	8	170	1375 F.C.
D	5 Cr, ½ Mo (Ti)	0.05	0.40	0.006	0.011	0.31	5.19	0.51	0.007	0.075	0.55 Ti	1–4 6–7	107 123	1375 F.C.
E	8 Cr, 1 Mo	0.099	0.38	0.013	0.015	0.32	8.21	1.06	...	...	0.14 Ni	8	142	1700 F.C.
F	8 Cr, 1 Mo (Cb)	0.09	0.47	0.015	0.011	0.34	8.20	1.00	...	...	0.55 Cb	8–9	143	1700 F.C.
G	12 Cr	0.086	0.43	0.018	0.009	0.39	12.52	...	...	...	0.09 Ni 0.03 Cu	6–8	252	1750 Q + 1150 A.C.
H	12 Cr, ½ Mo	0.08	0.37	0.022	0.021	0.22	12.14	0.49	...	...	0.19 Ni	5–8	231	1750 Q + 1220 A.C.
I[a]	12 Cr, ½ Mo (B)	0.07	0.38	0.020	0.031	0.20	11.94	0.52	...	...	0.0061 B 0.19 Ni	8–9	225	1750 Q + 1220 A.C.
J[a]	12 Cr, ½ Mo (Cb)	0.08	0.49	0.017	0.028	0.26	12.18	0.56	...	...	0.11 Cb 0.70 Ni	9–10	240	1750 Q + 1400 A.C.
K	17 Cr (430)	0.09	0.41	0.010	0.014	0.237	16.87	0.02	0.078	0.004	0.15 Ni	5–6	158	1425 A.C.

[a] Experimental steels.

TABLE II.—RESULTS OF TENSION TESTS[a] AT ROOM AND AT ELEVATED TEMPERATURES FOR STEELS E (8 Cr, 1 Mo) AND F (8 Cr, 1 Mo (Cb)).

Steel	Test Temperature, deg Fahr	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
E (8 Cr, 1 Mo)	75	31 200	75 000	31.0	66.1
	300	26 200	66 500	28.0	66.0
	500	24 700	62 000	25.5	65.5
	800	23 000	55 500	24.0	62.0
	1000	21 400	47 500	35.0	74.0
	1200	15 500	28 500	64.5	92.0
	1400	6 800	11 900	94.	98.5
F (8 Cr, 1 Mo (Cb))	75	34 800	71 400	33.5	74.9
	300	30 000	63 500	31.5	74.0
	500	28 500	60 500	27.5	75.0
	800	25 200	53 500	27.0	72.0
	1000	22 000	42 600	38.5	79.0
	1200	15 200	25 200	71.5	93.0
	1400	7 200	11 200	86.5	96.7

[a] Test specimens had a diameter of 0.5 in. and a gage length of 2 in. Average of duplicate tests at 75 and 1400 F but single tests at other temperatures.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 657.

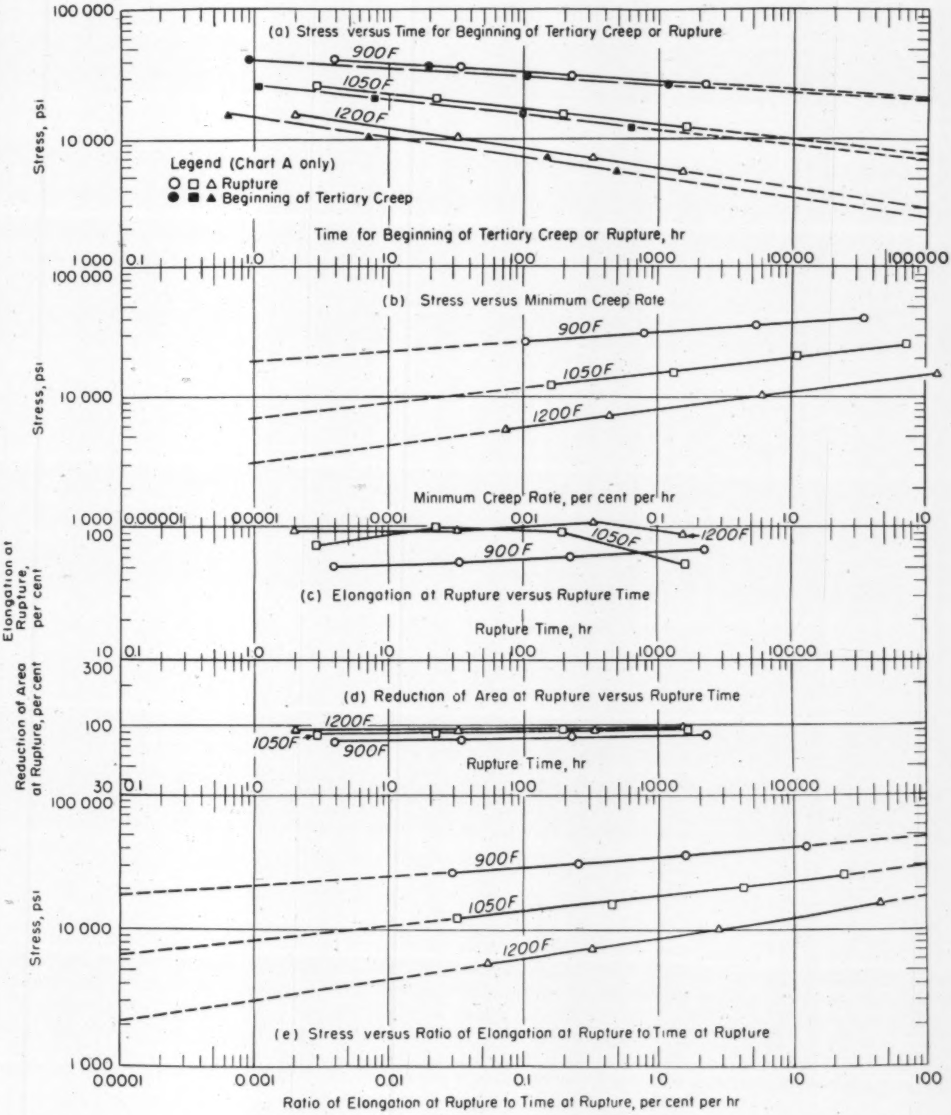

FIG. 1.—Properties of Steel A (5 Cr, 0.5 Mo).

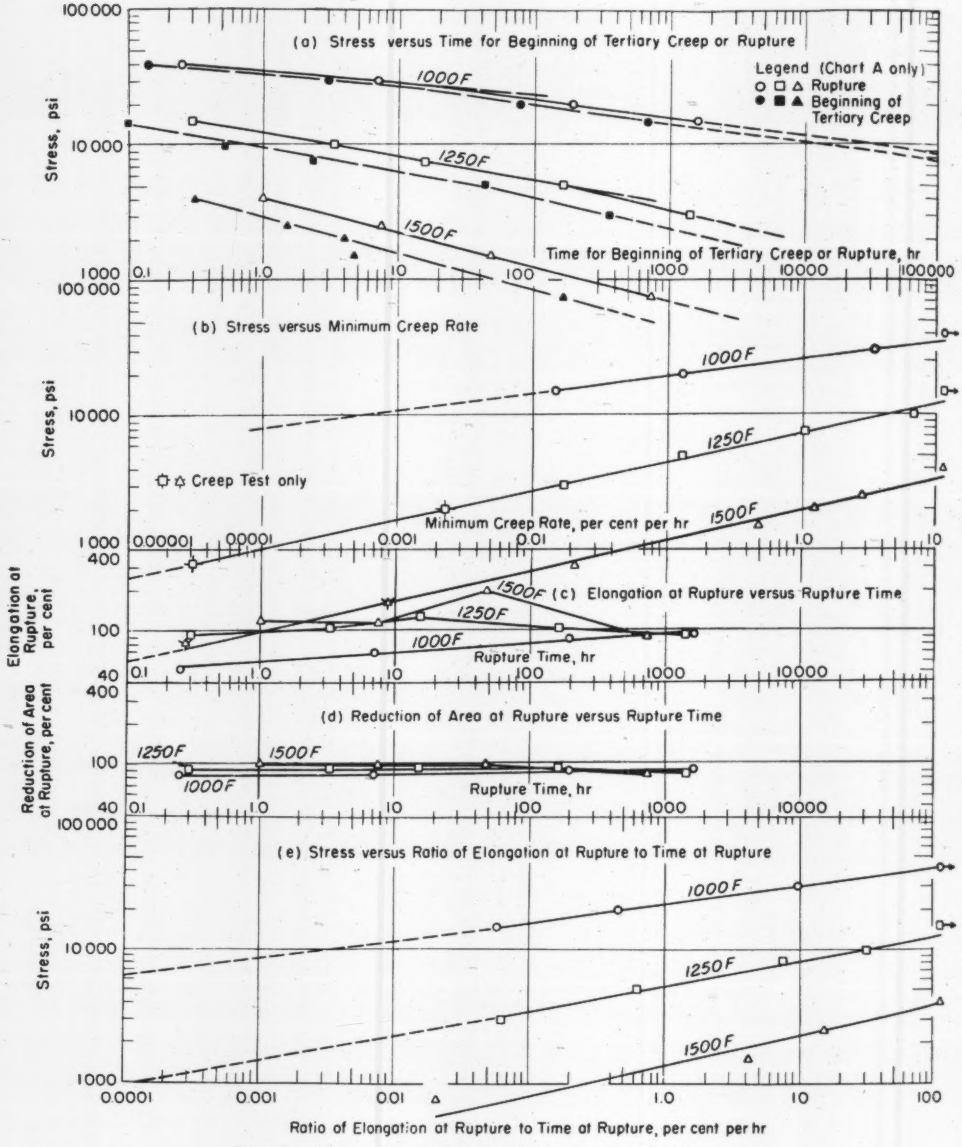

FIG. 2.—Properties of Steel B (5 Cr, 0.5 Mo (Si)).

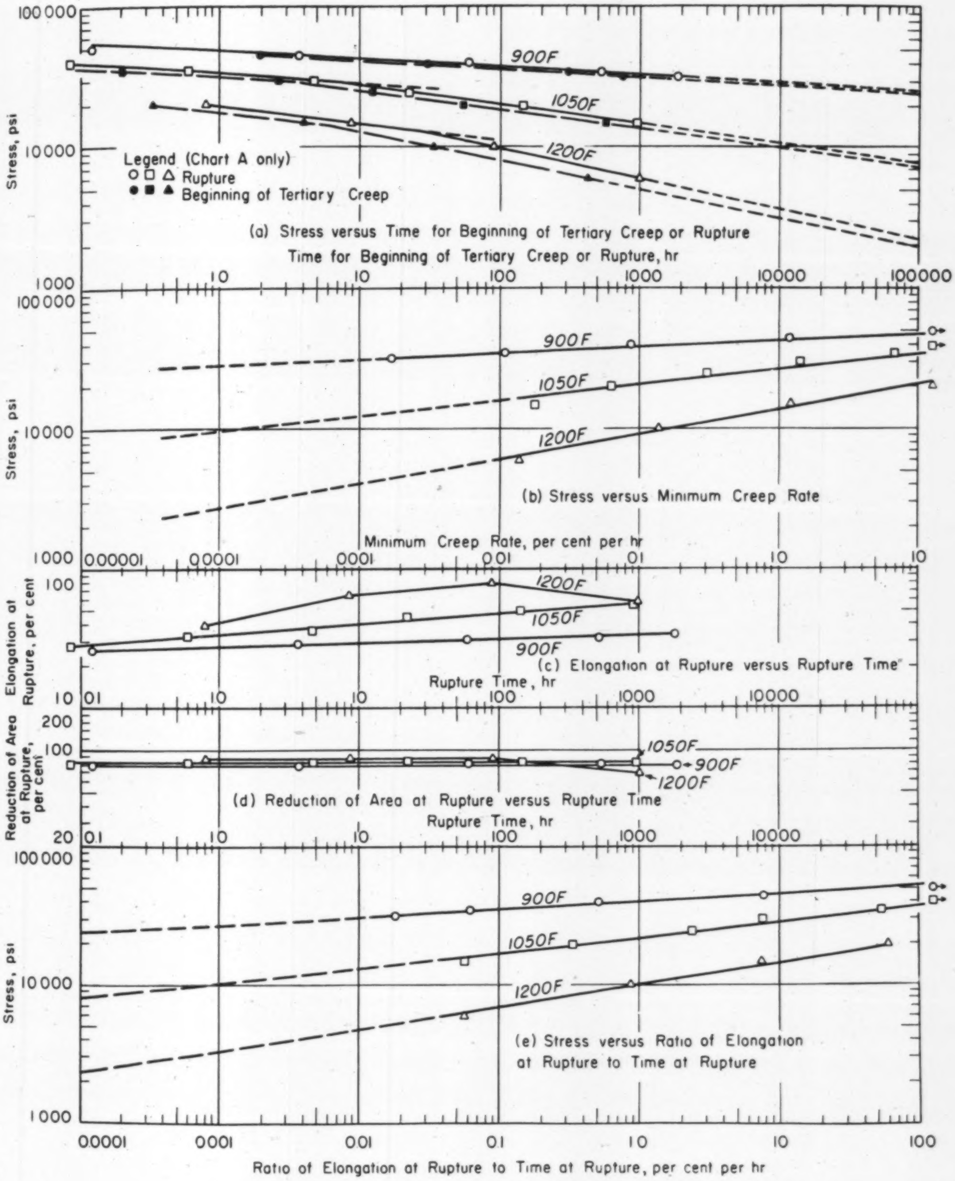

FIG. 3.—Properties of Steel C (5 Cr, 0.5 Mo (Cb)).

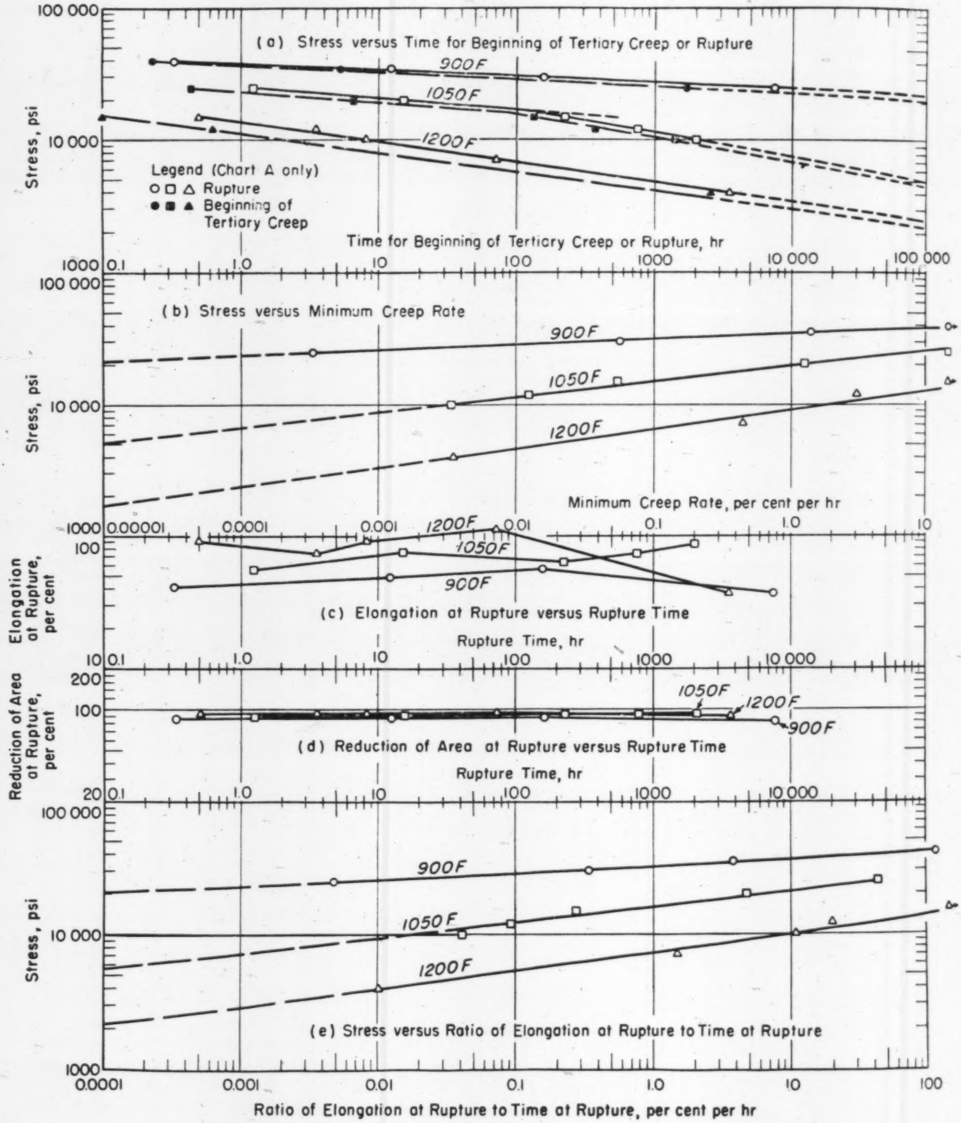

FIG. 4.—Properties of Steel D (5 Cr, 0.5 Mo (Ti)).

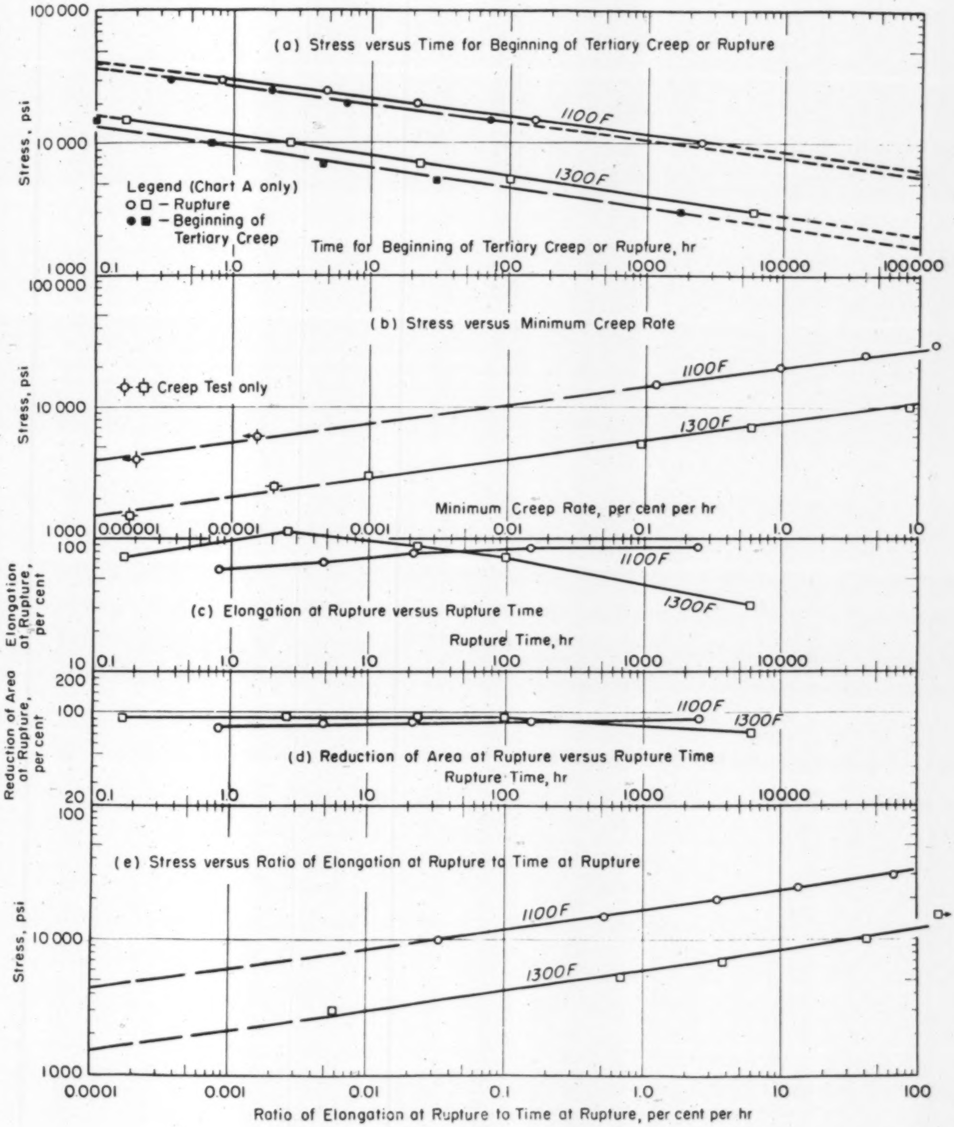

FIG. 5.—Properties of Steel E (8 Cr, 1 Mo).

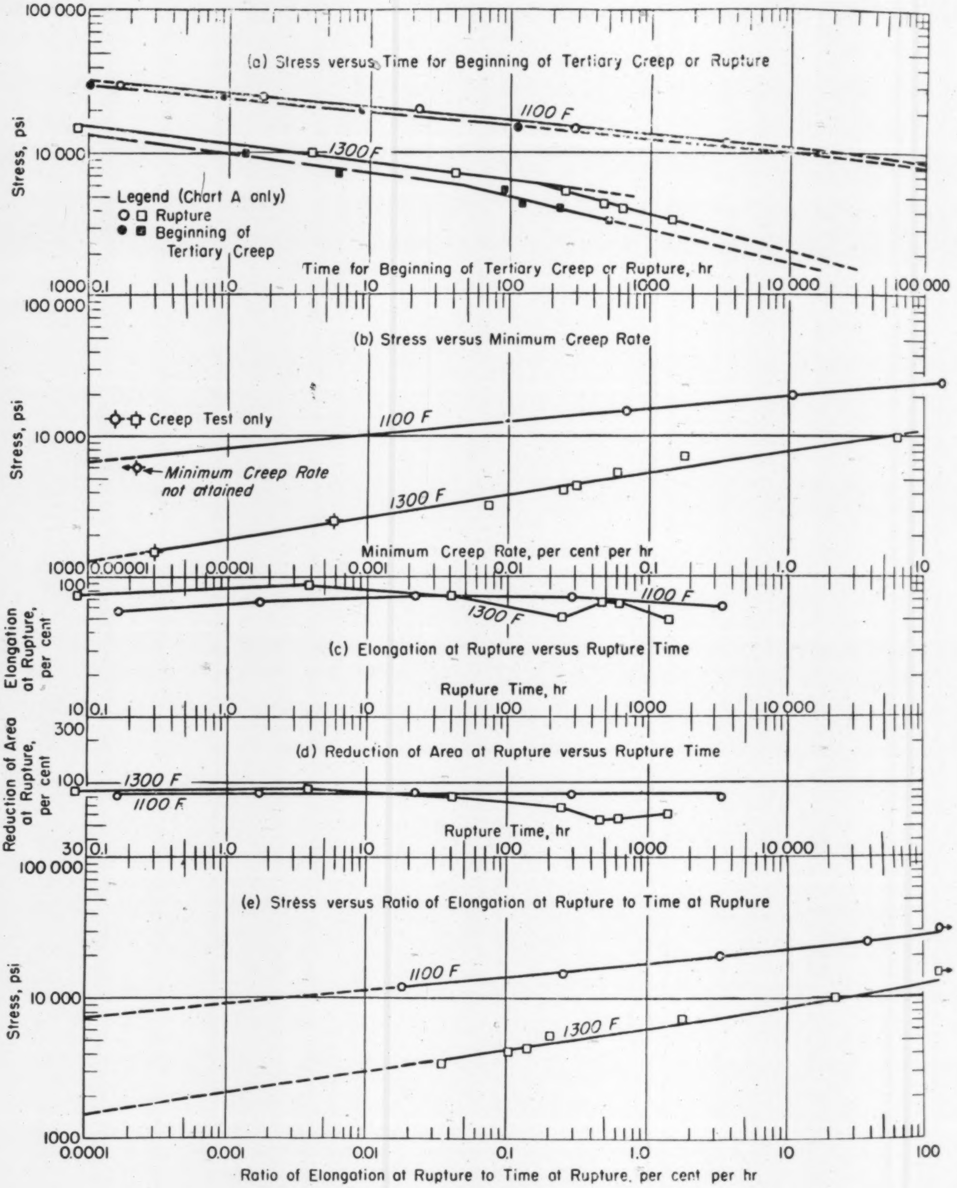

FIG. 6.—Properties of Steel F (8 Cr, 1 Mo (Cb)).

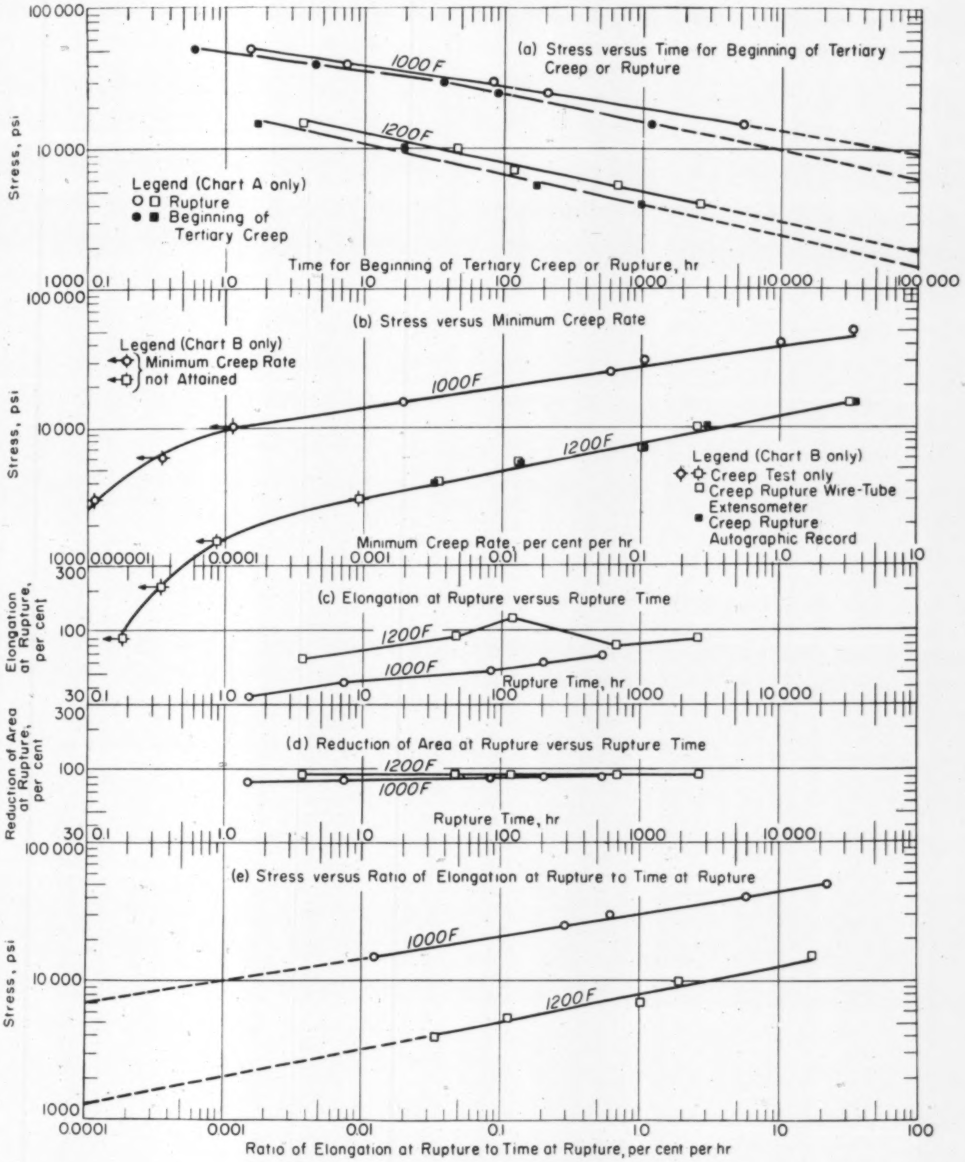

FIG. 7.—Properties of Steel G (12 Cr).

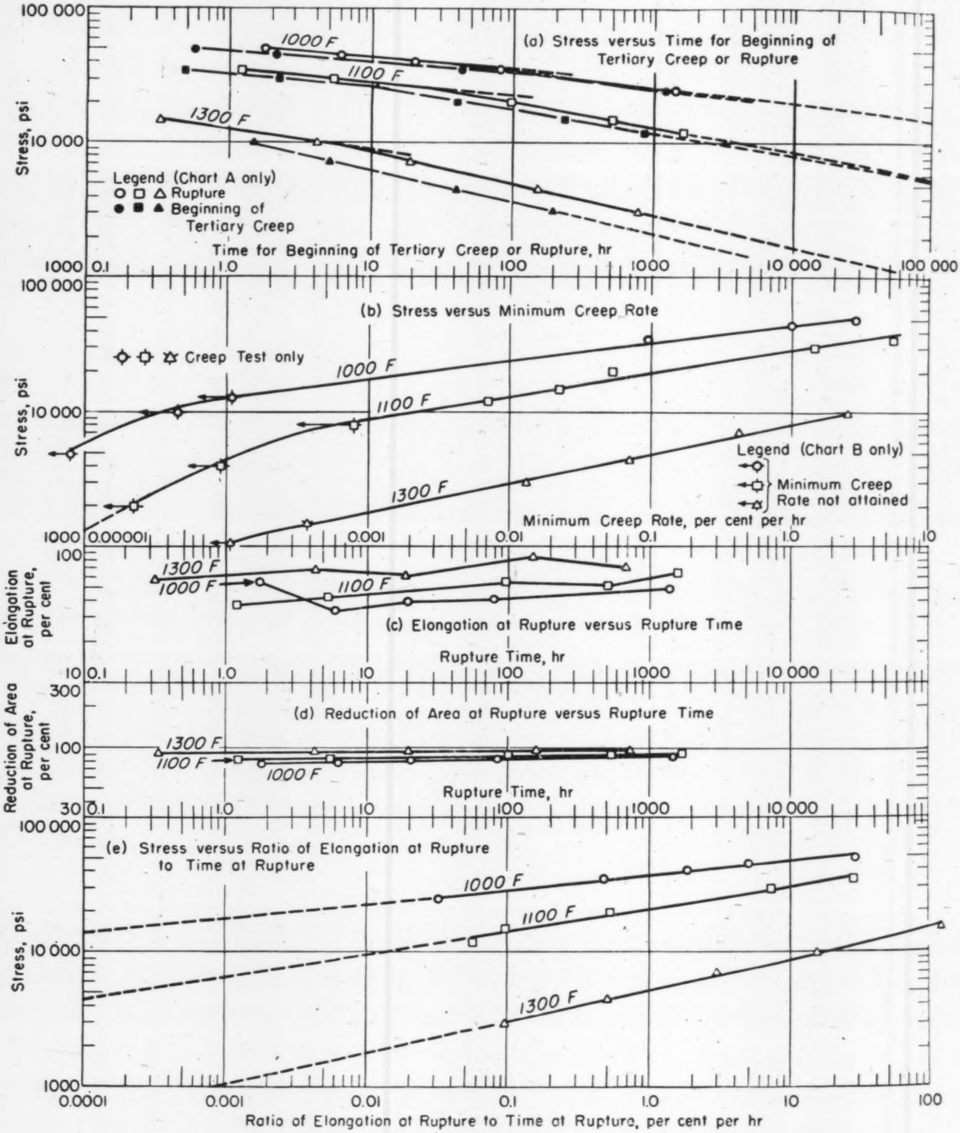

FIG. 8.—Properties of Steel H (12 Cr, 0.5 Mo).

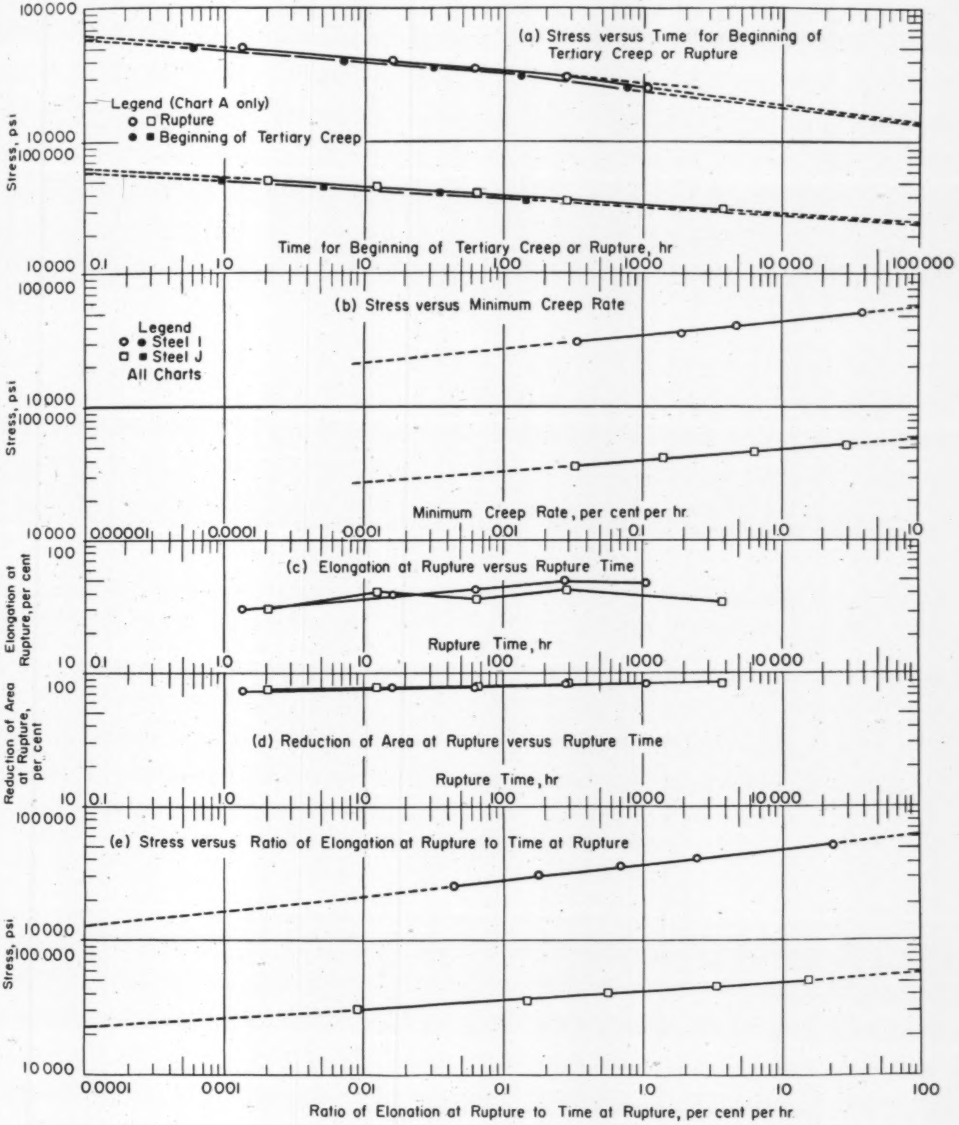

FIG. 9.—Properties of Steels I (12 Cr (Mo-B)) and J (12 Cr (Mo-Cb)) at 1000 F.

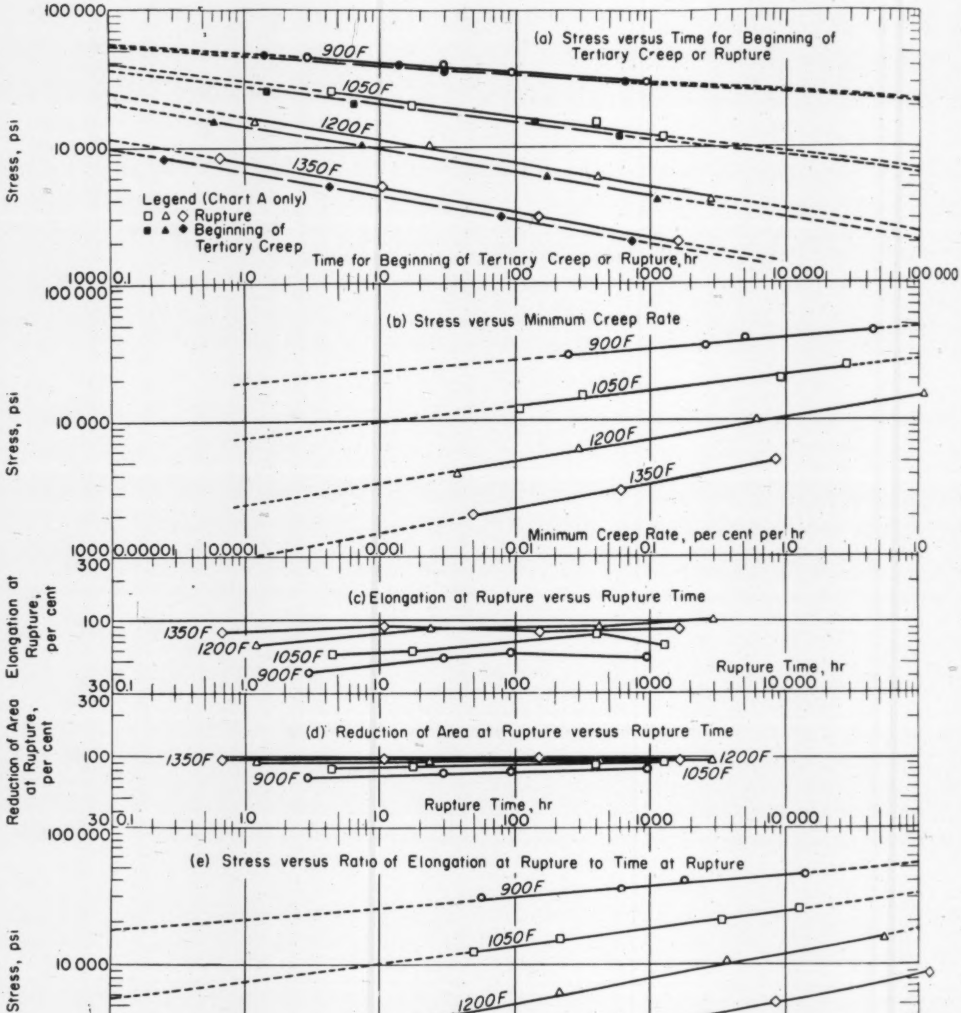

FIG. 10.—Properties of Steel K (17 Cr).

ment with the wire-tube extensometer. Illustrative of this good agreement are the data for the 12 Cr steel, shown in Fig. 7, wherein the creep rates obtained by both methods are shown.

Tension and impact tests were made in accordance with recognized testing procedures. Specimens for impact tests after creep were machined from standard ½-in. diameter creep specimens which necessitated the use of a ⅔ standard size specimen. This size Charpy keyhole-notch specimen was therefore used throughout the investigation. Room-temperature tension tests were made on some of the after-creep test specimens.

Specimens were examined before and after creep and creep-rupture tests to determine microstructural changes and to obtain any possible information about the mechanisms of flow and fracture during tests of this kind.

RESULTS AND DISCUSSION

In general, a time-extension (creep) curve consists of four parts: (1) initial deformation, comprising elastic strain and generally some plastic strain, which occurs immediately on application of load; (2) first-stage or primary creep, characterized by a decelerating rate of creep; (3) a second stage during which the creep rate is essentially constant (and a minimum); and (4) a third or accelerating-rate stage of creep which continues to fracture.

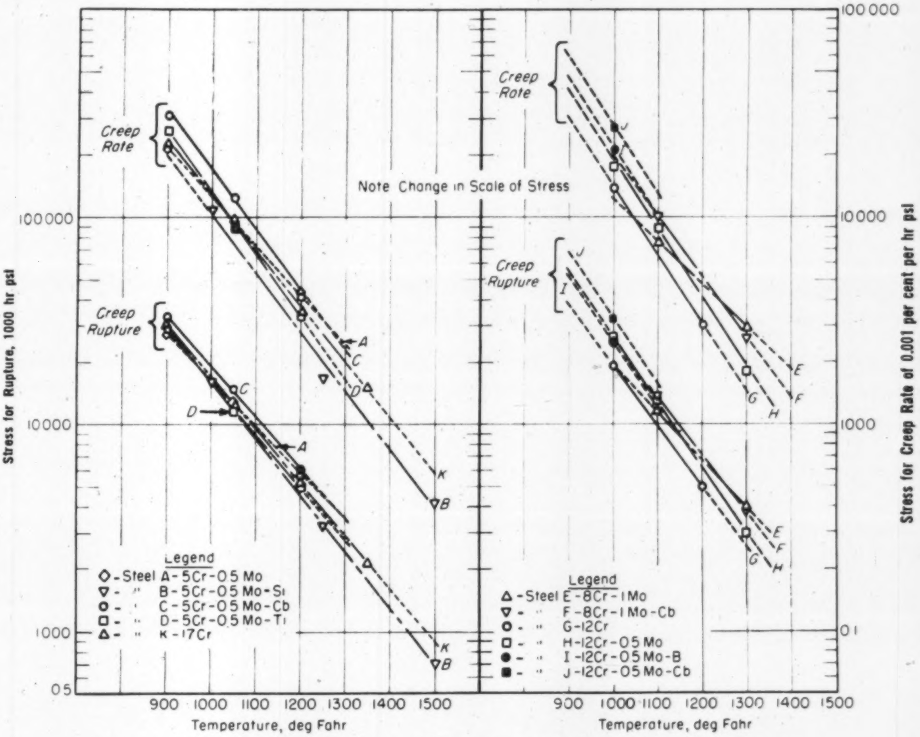

FIG. 11.—Dependence of Creep and Creep-Rupture Strength on Temperature.

As discussed in an earlier publication (3), relationships exist which enable the calculation of the total amount of creep at any time before the beginning of tertiary creep and also permit estimation of the total deformation to be expected at fracture. Total creep C_t up to any time t during minimum or

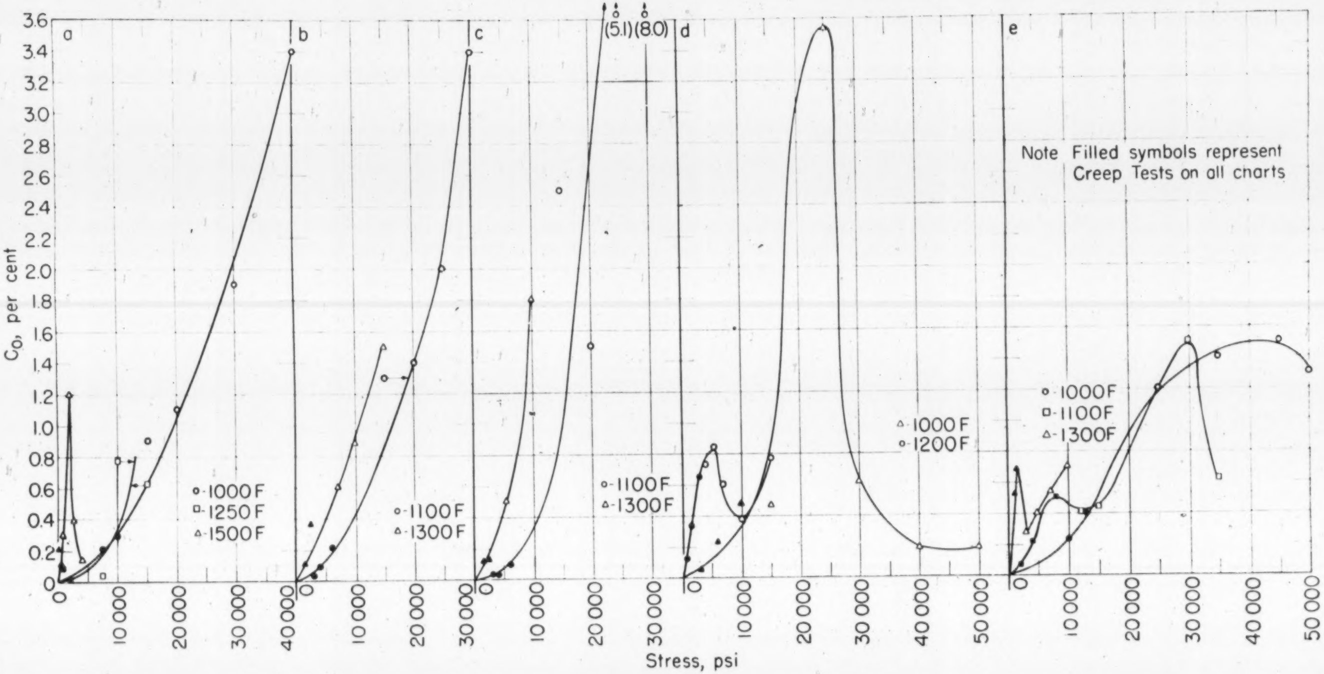

FIG. 12.—Dependence of Intercept C_0 on Stress.

(*a*) Steel B (5 Cr, 0.5 Mo (Si)).
(*b*) Steel E (8 Cr, 1 Mo).
(*c*) Steel F (8 Cr, 1 Mo (Cb)).
(*d*) Steel G (12 Cr).
(*e*) Steel H (12 Cr, 0.5 Mo).

constant rate of creep may be calculated by the following formula:

$$C_t = C_0 + \left(\frac{dC}{dt}\right)_m t \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where:

C_0 = intercept of the extrapolated minimum creep rate on the zero-time ordinate,

$\left(\frac{dC}{dt}\right)_m$ = minimum rate of creep, and

t = variable time.

This procedure can be applied only up to the beginning of tertiary or accelerating creep. The estimate of deformation at rupture is made on the basis of the observed linear relation on a log-log plot between stress and average rate of deformation, the latter being the quotient of the deformation at rupture by the time for rupture. Accordingly, the evaluation of the creep and rupture behavior of metals requires the experimental determination of the relations between stress and the several quantities: (1) minimum creep rate; (2) intercept C_0; (3) time for beginning of tertiary creep; (4) time for rupture; and (5) average creep rate. These relations, as far as the data are available, are shown in Figs. 1 to 10 and 12 of this paper; plots of elongation and reduction of area at fracture are also given in these charts.

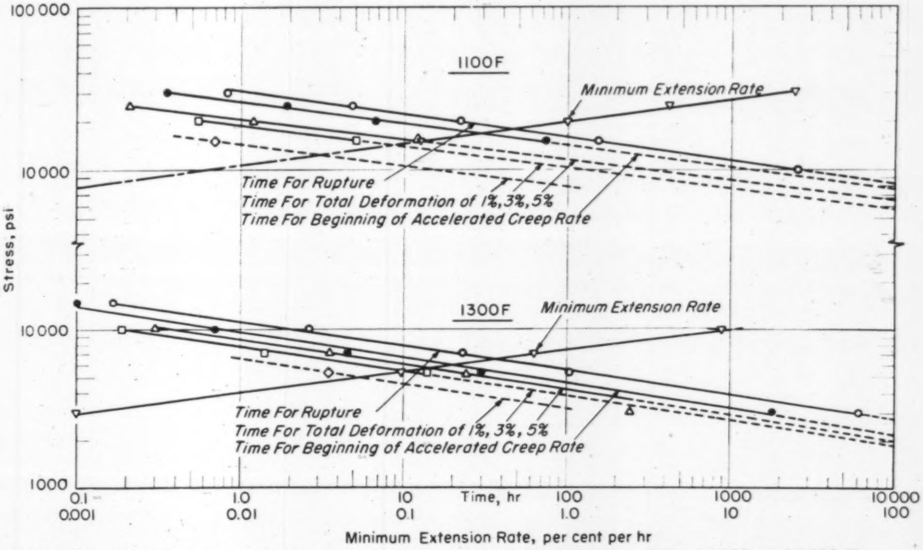

FIG. 13.—"Design Curves" for Creep to Rupture of 8 Cr, 1 Mo Steel E at 1100 and 1300 F.

In respect to the intercept C_0 data, most of the results for the present steels antedate our use of the wire-in-tube extensometer for the creep-rupture tests and are therefore only approximate, as explained earlier. Because of this, and also because some curious reversals were noted in several cases (which, however, are apparently real[3]), C_0 data are presented herein only for those steels for which creep, as well as creep-rupture, tests were made. Typical of the reversal noted in several instances is the curve

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[3] McVetty (8) has previously concluded that, although C_0 increases with stress at relatively low stress or temperature (according to a hyperbolic sine law), it passes through a maximum beyond which it decreases with further increase of stress.

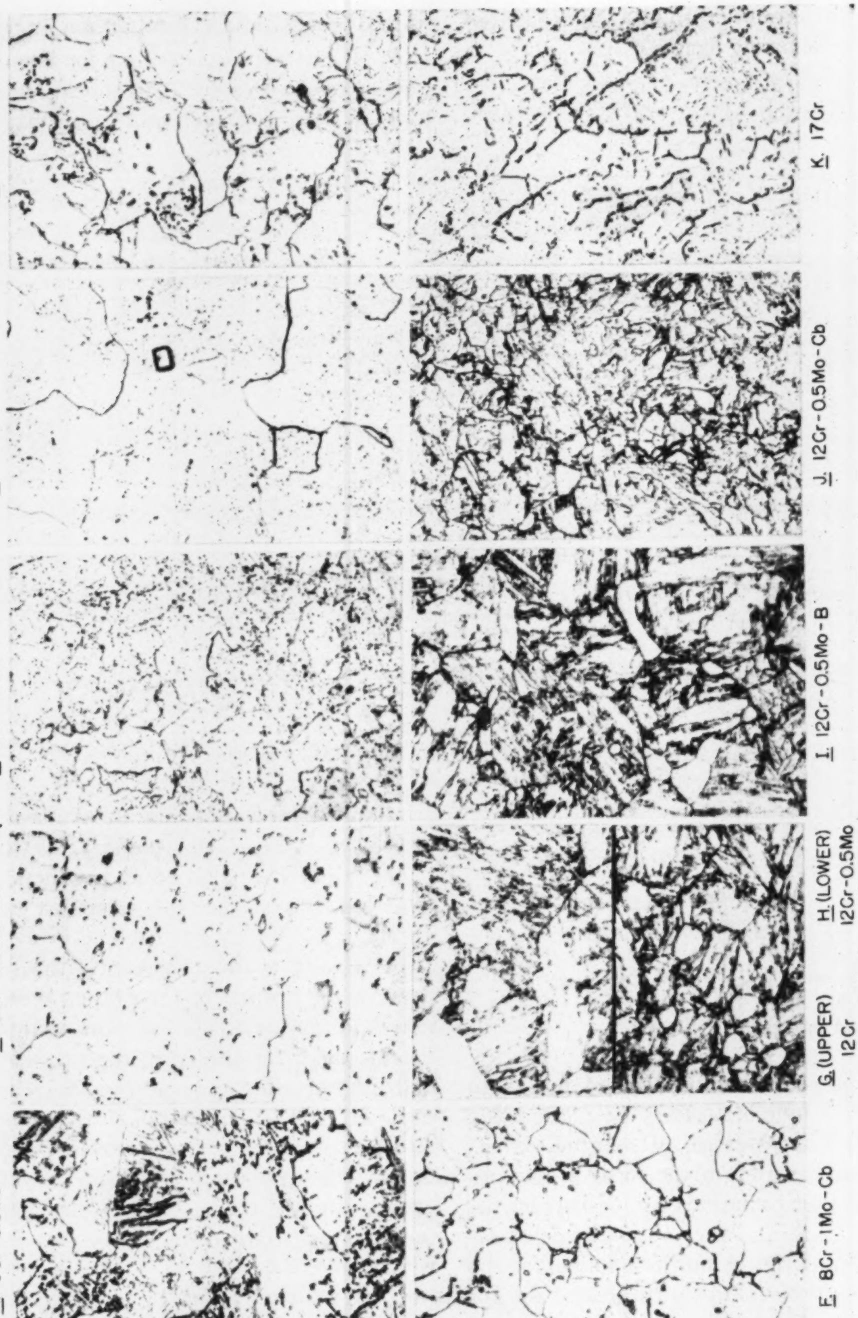
A. 5Cr-0.5Mo
B. 5Cr-0.5Mo-Si
C. 5Cr-0.5Mo-Cb
D. 5Cr-0.5Mo-Ti
E. 8Cr-1Mo
F. 8Cr-1Mo-Cb
G. (UPPER) 12Cr
H. (LOWER) 12Cr-0.5Mo
I. 12Cr-0.5Mo-B
J. 12Cr-0.5Mo-Cb
K. 17Cr

FIG. 14.—Steels A through K Prior to Test. Heat Treatment Listed in Table I. Picric-HCl Etch (× 1000). (Reduced to $^{7}/_{10}$ size in reproduction.)

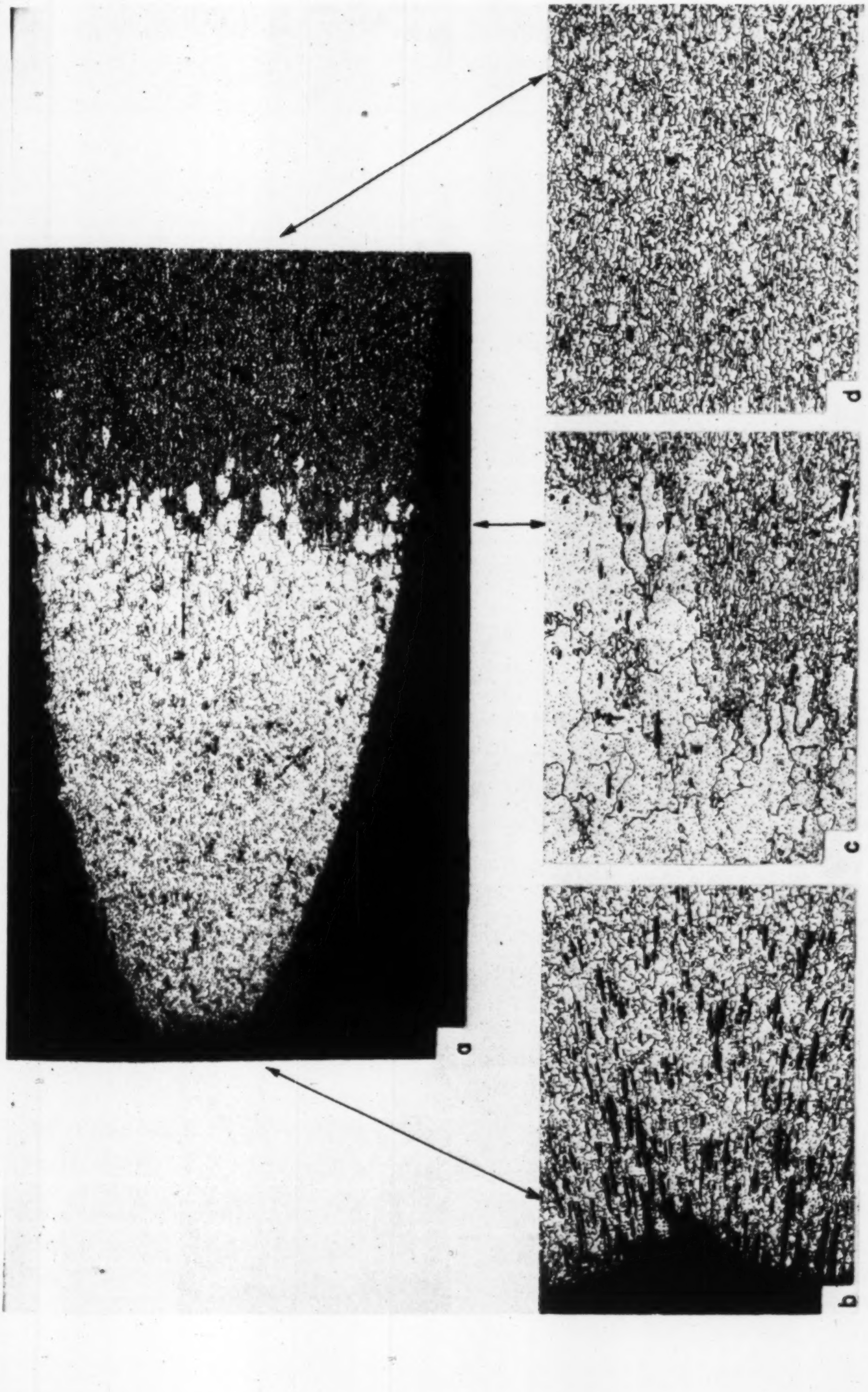

FIG. 15.—Longitudinal Sections of Creep-Rupture Specimen of Steel B (5 Cr, 0.5 Mo (Si)) Which Broke in 1.0 hr at 1500F. Magnification of (*a*) is 25×; (*b*), (*c*), (*d*) are 100×. (Reduced to $\frac{3}{4}$ size in reproduction.)

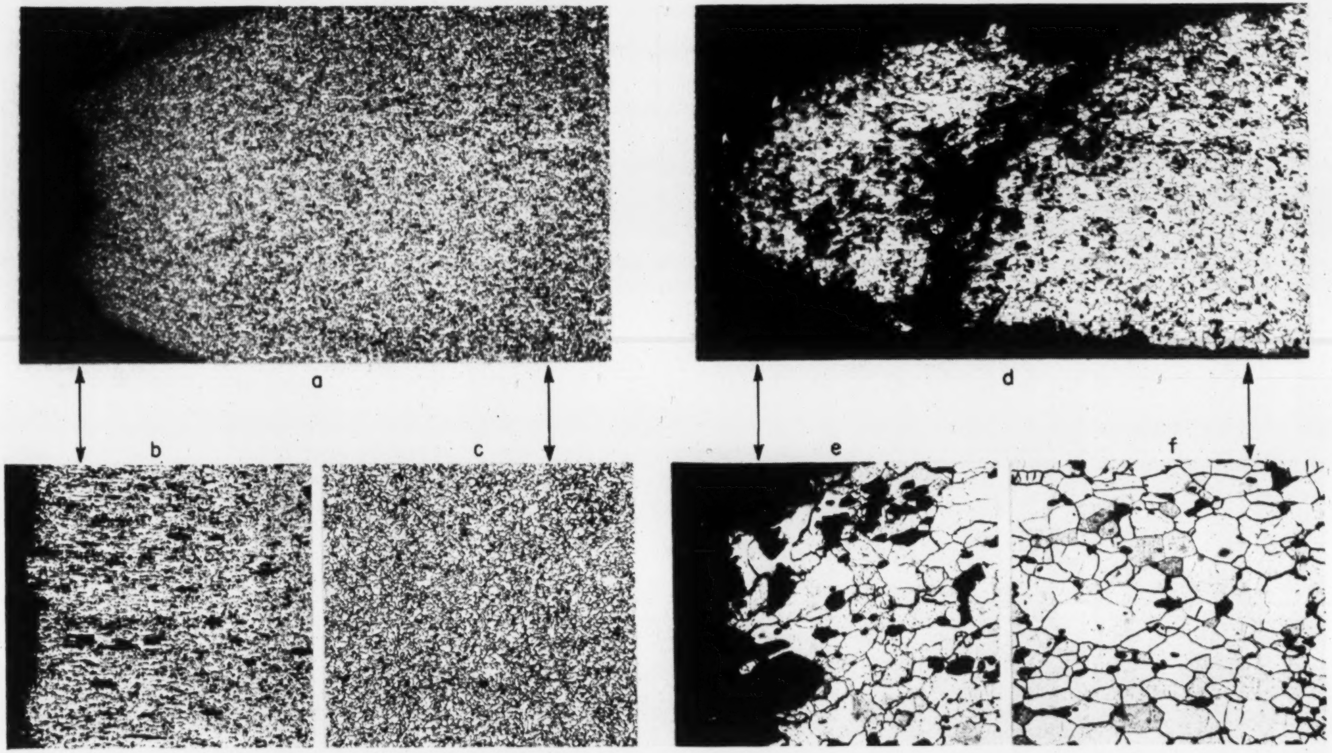

FIG. 16.—Longitudinal Sections of Creep-Rupture Specimens of Steel B (5 Cr, 0.5 Mo (Si))

(*a*), (*b*), (*c*) Portions of specimen that broke in 0.25 hr at 1000 F with 51 per cent elongation.
(*d*), (*e*), (*f*) Portions of specimen that broke in 743 hr at 1500 F with 90 per cent elongation.
Magnifications of (*a*) and (*d*) are 25×; (*b*,) (*c*), (*e*), and (*f*) are 100×. (Reduced to ⅔ size in reproduction.)

for 1500 F in Fig. 12(*a*), confirmed in this case by wire-tube extensometer measurements and also by the smoothness of the curve. It is also true, of course, that the rupture tests were generally made at such large stresses, relative to actual working stresses, that the associated C_0 values are of little practical interest in themselves (because the irregular relation precludes extrapolation) unless supplemented by tests more nearly of the magnitude that might be used in service.

Of interest to the design engineer is the relationship between stress and time for a specified total elongation. Data of this type are shown for steel E, 8 Cr, 1 Mo, in the "design chart," Fig. 13. Similar plots can be made for the other steels considered in this report, within the limitations of the data.

The empirical treatment of the data, as given in this report, is considered to be more worth while than attempting to fit the data to functional relations among the variables. Because the microstructure changes during tests at elevated temperatures and because structure and mechanical properties are interdependent, it is difficult, if not impossible in many instances, to generalize on behavior when a continual variation in structure is taking place.

Microstructures of the steels prior to test are shown in Fig. 14. The two important structural alterations which occurred during test (or immediately afterwards) were spheroidization and recrystallization; these changes are discussed in detail later. Some examples of recrystallization during creep-rupture tests are shown in Figs. 15 and 16. Although the 5 Cr, ½ Mo (Si) alloy may have approached the lower critical temperature (A_1) when tested at 1500 F, no evidence was found which indicated that this temperature had been exceeded. Previous unpublished work on the identical steel by R. F. Miller at this laboratory had shown no austenite after ¼ hr at 1550 F and only 5 per cent austenite after ¼ hr at 1650 F.

TABLE III.—SUMMARY OF CREEP-RUPTURE TEST RESULTS.

Steel	Type	Initial Treatment, deg Fahr	Stress for Rupture in 1000 hr, psi								
			900 F	1000 F	1050 F	1100 F	1200 F	1250 F	1300 F	1350 F	1500 F
A	5 Cr, ½ Mo	1550, F.C.	27 200	16 000[a]	12 600	9 600[a]	5 800	4 400[b]	3 400[b]	...	...
B	5 Cr, ½ Mo(Si)	1600, F.C.	30 000[b]	16 000	11 600[a]	8 500[a]	4 500[a]	3 200	2 400[a]	1 800[a]	700
C	5 Cr, ½ Mo(Cb)	1375, F.C.	33 000	19 000[a]	14 800	10 700[a]	6 000	4 600[b]	3 400[b]	...	...
D	5 Cr, ½ Mo(Ti)	1375, F.C.	28 300	16 000[a]	11 400	8 800[a]	4 900	3 700[b]	...	...	...
E	8 Cr, 1 Mo	1700, F.C.	...	19 000[b]	14 600[b]	11 300	6 600[a]	5 000[a]	3 900	3 000[b]	...
F	8 Cr, 1 Mo(Cb)	1700, F.C.	...	...	18 300[b]	13 500	7 000[a]	5 000[a]	3 700	2 600[b]	...
G	12 Cr	1750 Q + 1150 A.C.	37 000[b]	19 000	13 700[a]	9 800[a]	4 900	3 500[b]	2 500[b]	...	...
H	12 Cr, ½ Mo	1750 Q + 1220 A.C.	54 000[b]	26 000	18 000[a]	13 000	6 000[a]	4 200[a]	2 900	2 000[b]	...
I	12 Cr, ½ Mo(B)	1750 Q + 1220 A.C.	50 000[b]	25 000	17 000[b]	12 000[b]	...	...	...	...	...
J	12 Cr, ½ Mo(Cb)	1750 Q + 1400 A.C.	66 000[b]	32 500	22 000[b]	15 000[b]	...	...	...	...	...
K	17 Cr	1425 A.C.	29 500	16 000[a]	12 000	9 000[a]	5 000	3 800[a]	2 800[a]	2 100	880[b]

[a] Interpolated from Fig. 11.
[b] Extrapolated from Fig. 11.

MECHANICAL TESTS

Creep and creep-to-rupture test results are shown graphically in Figs. 1 to 10 and are summarized in Tables III, IV, and V. The influence of temperature on the creep strength (1 per cent per 1000 hr) and creep rupture strength (in 1000 hr) of these steels is shown in Fig. 11. The methods of plotting were discussed in detail in earlier publications (**1,3**).

Rupture Time:[4]

The creep-rupture test results are

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[4] The comparisons in this and subsequent sections refer only to the specific steels and heat treatments studied. To what extent they are generally true is not known.

shown in Figs. 1 to 10 by plots of log initial stress *versus* log rupture time. The data in this form are characterized by a single straight line or by two intersecting straight lines, depending on the steel, test temperature, and rupture time. Straight line variations such as these permit interpolation and limited extrapolation with a reasonable degree of confidence as discussed previously (**3**, **6**). Similarly, straight line variations of temperature *versus* log initial stress for rupture in 1000 hr permit interpolation of these data to intermediate temperatures, Fig. 11. Employing both of these plots, the data of Table III were obtained. With a minor exception, the rupture values at the test temperatures were obtained by interpolation. The limit of extrapolation to other temperatures did not exceed 150 F and was done only to permit comparison of different grades. Attention is directed especially to the fact that in the case of steels I and J, tested only at 1000 F, it was also necessary to assume that the slope of the temperature plot is parallel to that for plain 12 Cr, $\frac{1}{2}$ Mo steel H.

TABLE IV.—SUMMARY OF MINIMUM CREEP RATES FROM CREEP AND CREEP-RUPTURE TEST RESULTS.

Steel	Type	900 F	1000 F	1050 F	1100 F	1200 F	1250 F	1300 F	1350 F	1500 F
	Stress for Creep Rate of 0.001 per cent per hr, psi									
A	5 Cr, ½ Mo	22 000	12 200^{a}	8 800	7 100^{a}	4 200	3 200^{b}	2 400^{b}	...	...
B	5 Cr, ½ Mo(Si)	19 000^{b}	10 800	7 300^{a}	5 200^{a}	2 700^{a}	1 650	1 400^{a}	1 000^{a}	410
C	5 Cr, ½ Mo(Cb)	31 000	17 000^{a}	12 400	8 600^{a}	4 100	3 100^{b}	2 200^{b}	...	...
D	5 Cr, ½ Mo(Ti)	26 000	13 000^{a}	8 800	6 400^{a}	3 300	2 300^{b}	1 600^{b}	...	...
E	8 Cr, 1 Mo	...	...	9 500^{b}	7 500	4 600^{a}	3 700^{a}	2 900	2 300^{b}	...
F	8 Cr, 1 Mo(Cb)	...	...	14 000^{b}	10 000	5 100^{a}	3 600^{a}	2 600	1 900^{b}	...
G	12 Cr	29 000^{b}	13 800	9 400^{a}	6 400^{a}	3 000	2 000^{b}	1 400^{b}	...	...
H	12 Cr, ½ Mo	39 000^{b}	17 300	12 500^{a}	8 800	4 000^{a}	2 700^{a}	1 800	1 300^{b}	...
I	12 Cr, ½ Mo(B)	45 000^{b}	21 000	14 800^{b}	10 000^{b}	...	...	...	...	...
J	12 Cr, ½ Mo(Cb)	57 000^{b}	27 000	18 500^{b}	12 600^{b}	...	...	...	...	...
K	17 Cr	22 500	12 500^{a}	9 700	6 700^{a}	3 400	2 700^{a}	2 000^{a}	1 500	570^{b}
	Stress for Creep Rate of 0.0001 per cent per hr, psi									
A	5 Cr, ½ Mo	18 500	10 300^{a}	6 700	5 300^{a}	3 000	2 100^{b}			
B	5 Cr, ½ Mo(Si)	15 000^{b}	8 000	5 200^{a}	3 600^{a}	1 800^{a}	1 000	900^{a}	620^{a}	240
C	5 Cr, ½ Mo(Cb)	28 000	13 500^{a}	9 700	5 300^{a}	2 700	2 000^{b}	...	...	...
D	5 Cr, ½ Mo(Ti)	23 500	10 500^{a}	6 700	4 900^{a}	2 300	1 100^{b}	...	...	...
E	8 Cr, 1 Mo	...	...	...	5 400	...	...	2 100	...	...
F	8 Cr, 1 Mo(Cb)	...	...	11 900^{b}	8 100	3 800^{a}	2 600^{a}	1 800	1 200^{b}	...
G	12 Cr	24 000^{b}	9 600	6 100^{a}	3 900^{a}	1 560	970^{b}	620^{b}	...	...
H	12 Cr, ½ Mo	26 000^{b}	12 800	7 800^{a}	4 400	2 300^{a}	1 500^{a}	1 100	...	...

[a] Interpolated from strength-temperature curves.
[b] Extrapolated from strength-temperature curves.

TABLE V.—ELONGATION AT RUPTURE IN 10,000 hr.

Steel	Type	Initial Treatment, deg Fahr	Extrapolated per cent Elongation at Rupture in 10,000 hr							
			900 F	1000 F	1050 F	1100 F	1200 F	1250 F	1300 F	1350 F
A	5 Cr, ½ Mo	1550, F.C.	80	...	55	...	100	...	...	...
B	5 Cr, ½ Mo(Si)	1600, F.C.	...	110	...	...	...	40	...	...
C	5 Cr, ½ Mo(Cb)	1375, F.C.	30	...	18	...	17	...	...	...
D	5 Cr, ½ Mo(Ti)	1375, F.C.	60	...	15	...	36	...	...	...
E	8 Cr, 1 Mo	1700, F.C.	...	...	...	70	...	...	60	...
F	8 Cr, 1 Mo(Cb)	1700, F.C.	...	...	...	70	...	...	10	...
G	12 Cr	1750, O.Q. + 1150 A.C.	...	66	...	...	72	...	...	...
H	12 Cr(Mo)	1750, O.Q. + 1220 A.C.	...	30	...	50	...	...	80	...
I	12 Cr(Mo, B)	1750 Q + 1220 A.C.	...	27	...	...	...	...	...	...
J	12 Cr(Mo, Cb)	1750 Q + 1400 A.C.	...	35	...	...	...	...	...	...
K	17 Cr	1425, A.C.	110	...	45	...	115	...	...	80

Whereas at lower temperatures creep-rupture strength of any grade was generally increased by alloy addition, the opposite was true in several cases at the higher temperatures. Steel J (12 Cr, $\frac{1}{2}$ Mo (Cb)) had the highest creep-rupture strength at lower test temperatures, but unfortunately tests at higher temperatures were not made for this steel. Creep-rupture strength at 1000 F ranged from 32,500 psi for steel J (12 Cr, $\frac{1}{2}$ Mo (Cb)) to 16,000 psi for four steels, K (17 Cr), A (5 Cr, $\frac{1}{2}$ Mo), B (5 Cr, $\frac{1}{2}$ Mo (Si)), and D (5 Cr, $\frac{1}{2}$ Mo (Ti)).

Of the 5 Cr, $\frac{1}{2}$ Mo group, steel C (5 Cr, $\frac{1}{2}$ Mo (Cb)) is the strongest at lower temperatures (900 to 1250 F) but above 1300 F, steel A (5 Cr, $\frac{1}{2}$ Mo) has the highest strength of the group. Addition of silicon or titanium evidently decreases creep-rupture strength in this grade as indicated by the lower strength of steels D (5 Cr, $\frac{1}{2}$ Mo (Ti)) and B (5 Cr, $\frac{1}{2}$ Mo (Si)) at all except the lower test temperatures.

The two 8 Cr, 1 Mo steels showed relatively little difference in strength at 1250 F and above, steel E (8 Cr, 1 Mo) being slightly stronger, but below 1250 F steel F (8 Cr, 1 Mo (Cb)) was stronger.

In the 12 Cr group, steel J (12 Cr (Mo-Cb)) had the highest creep-rupture strength at 1000 F; the strengths ranged from 32,500 psi for steel J (12 Cr (Mo-Cb)) to 19,000 psi for steel G (12 Cr). The boron addition appears to have had little effect on the creep-rupture strength of this type of steel, as indicated by these limited tests.

Steel K (17 Cr) was relatively weak in creep-rupture, being comparable to steels B and D, which were the weakest of the entire group studied.

From the limited tests of this study, the general effect of the addition of specific alloying elements to high chromium-molybdenum steels on creep-rupture strength is indicated to be: (*a*) columbium increases strength at temperatures up to 1300 F above which it is detrimental; (*b*) silicon and titanium decrease strength at least at the higher temperatures investigated, and (*c*) boron has no appreciable effect. It is to be remembered, however, that variations in initial heat treatment or chemical composition might markedly affect the creep-rupture strength, and only the commercial heat treatments and one heat of each steel were investigated in this study. Thus the general superiority of the 12 Cr grades at the lower temperatures may be associated with the higher initial, room temperature, hardness level.

Minimum Creep Rate:

Plots of minimum creep rate *versus* stress, on log-log coordinates, for all of the steels are shown in Figs. 1 to 10, and a summary of these data expressed as the stress to produce a creep rate of 0.001 or 0.0001 per cent per hr is given in Table IV. The effect of test temperature on creep strength is given in Fig. 11. Essentially straight line variations are shown for plots of log of stress to produce a minimum creep rate of 0.001 per cent per hr (1 per cent per 1000 hr)[5] *versus* temperature (exponential variation); stress to produce a minimum creep rate of 1 per cent per 10,000 hr, Table IV, was obtained for some of the steels from a similar graph. The data were taken from both creep and creep-rupture test curves of extension *versus* time for all of the stresses and temperatures investigated. Extrapolations were made in some cases for purposes of preparing Table IV. Extrapolation of creep rate did not exceed 2 cycles, whereas the extrapolation to temperatures other than those tested did not exceed 150 F.

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[5] Creep strength is usually reported for minimum creep rates of 0.0001 per cent or 0.00001 per cent per hr; however, to obtain these data from our results would involve questionable extrapolations in some cases The 0.001 per cent per hr rate may be used to compare the steels.

The relative creep strength of the steels is, in general, similar to that of their creep-rupture strength, steel J (12 Cr, $\frac{1}{2}$ Mo (Cb)) having the highest and steels A (5 Cr, $\frac{1}{2}$ Mo), B (5 Cr, $\frac{1}{2}$ Mo (Si)), D (5 Cr, $\frac{1}{2}$ Mo (Ti)), and J (17 Cr) the lowest creep strength at the lower test temperatures (900 to 1050 F).

The 5 Cr, $\frac{1}{2}$ Mo group of steels shows the same trends in creep strength as in creep-rupture strength, except that the temperature at which the columbium-bearing steel, C, decreases in relative strength as compared to the straight chromium-steel, A, is lower (1200 F). At 900 F the creep strength for a minimum creep rate of 0.001 per cent per hr of the steels was: steel C (5 Cr, $\frac{1}{2}$ Mo (Cb)), 31,000 psi; steel D (5 Cr, $\frac{1}{2}$ Mo (Ti)), 26,000 psi; steel A (5 Cr, $\frac{1}{2}$ Mo), 22,000 psi; and steel B (5 Cr, $\frac{1}{2}$ Mo (Si)), 19,000 psi. Creep strength, from Fig. 11, at 1300 F was: steel A, 2400 psi; steel C, 2200 psi; steel D, 1600 psi, and steel B, 1400 psi. Similarly, the 8 Cr, 1 Mo steels follow the same trends, that is, creep strength of steel F (8 Cr, 1 Mo (Cb)) is higher than for steel E (8 Cr, 1 Mo) up to 1200 F, above which the reverse is true. At 1100 F, steel F had a creep strength (0.001 per cent per hr) of 10,000 psi and steel E 7500 psi, whereas at 1300 F the creep strength was 2600 and 2900 psi, respectively.

Of the 12 Cr group tested at 1000 F, steel J (12 Cr, $\frac{1}{2}$ Mo (Cb)) had a creep strength (0.001 per cent per hr) of 27,000 psi, whereas the values for steels I (12 Cr, $\frac{1}{2}$ Mo (B)), H (12 Cr, $\frac{1}{2}$ Mo), and G (12 Cr) were 21,000, 17,300 and 12,500 psi, respectively. Unfortunately, as for creep-rupture properties, results of tests at temperatures other than 1000 F were not available for steels I and J to determine the true slope of the lines shown in Fig. 11 (*b*). The 12 Cr, $\frac{1}{2}$ Mo steels with boron and columbium additions, especially the latter, had higher creep strength than the steel without these additions.

Steel K (17 Cr) at the lower test temperatures (900 to 1300 F) had a relatively low creep strength (0.001 per cent per hr), 22,500 psi at 900 F, but at the higher temperatures it compared favorably with the rest of the steels (1500 psi at 1350 F).

Deviations from a straight line for the log-log plot of stress *versus* minimum creep rate were found at low stresses (low creep rates) for steels G (12 Cr) and H (12 Cr, $\frac{1}{2}$ Mo), Figs. 7 and 8. This behavior was previously reported by Nadai and McVetty (**4**, **5**), who observed that the relation between stress and minimum creep rate is more accurately given for some materials by a hyperbolic sine function rather than the usual power function of a log-log plot,[6] especially for extrapolation to low creep rates. However, we have not hitherto observed such pronounced deviations from a straight line at rates as fast as those for which deviation is observed for these 12 Cr steels; this unusual behavior is presumably characteristic of the material tested and possibly of the prior heat treatment. In view of the behavior of steels G (12 Cr) and H (12 Cr, $\frac{1}{2}$ Mo), Figs. 7 and 8, extrapolation to creep rates less than 0.001 per cent per hr has not been made for steels I (12 Cr, $\frac{1}{2}$ Mo (B)), J (12 Cr, $\frac{1}{2}$ Mo (Cb)), or K (17 Cr), Figs. 9 and 10, as these steels may possibly behave like steels G and H.

Beginning of Tertiary Creep:

The time for beginning of tertiary or accelerated creep is shown as a function of initial stress for each steel and test temperature on log-log plots in (*a*) of Figs. 1 to 10; the relationship parallels that for time to rupture.

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[6] A feature of the hyperbolic sine law is that a power function defines the tangent to the hyperbolic sine curve and under certain conditions is a close approximation to it.

It is interesting to note that the time of third stage creep, that is, the difference between rupture time and time for beginning of tertiary creep, Fig. 2, is exceptionally long for steel B (5 Cr, $\frac{1}{2}$ Mo (Si)) tested at 1250 and 1500 F. This steel exhibited recrystallization during or possibly immediately after test at these temperatures (see later discussion) as well as outstandingly high elongation and reduction of area. It is interesting to note further that, as is often the case, most of the deformation occurred during the third stage. Thus, steel B tested at 1500 F with an initial stress of 2000 psi had an elongation of only 6.2 per cent at the beginning of tertiary creep but a final elongation of 136 per cent.

Intercept Deformation, C_0:

Variation with stress of C_0, intercept of the extrapolated minimum creep rate on the zero time ordinate, is shown in Fig. 12 for the five steels for which data from both creep and creep-rupture tests were obtained, as explained earlier.

Whereas C_0 data for other steels previously reported (**3**, **7**) showed a fairly consistent variation, C_0 increasing with increasing stress, the data reported herein show reversals in several instances. Similar reversal has been reported by McVetty (**8**) who found in some cases a maximum value with increasing stress beyond which C_0 decreases. Intercept data reported by Manjoine (**9**) also indicate an irregular relationship in some instances.

As the quantity C_0 is a function of deformation on application of load, deformation during first stage, and slope of minimum creep rate line, it is obvious that the form of the time-extension curve, which is influenced by many factors, is an important consideration in the determination of C_0.

Elongation and Reduction of Area at Fracture:

As a need existed for a correlation between elongation at rupture and time to rupture, which would permit extrapolation to longer rupture times, a method was found (**3**) which would give the desired data by employing the plots of log stress *versus* log rupture time ((*a*) of Figs. 1 to 10) and of log stress *versus* log ratio of elongation at rupture to time at rupture ((*e*) of Figs. 1 to 10), both of which have a linear relation. The procedure is to obtain the stress corresponding to the rupture time in question from (*a*), find the average creep rate for this stress in (*e*), and multiply the average creep rate by the rupture time to get the elongation at rupture. Extrapolated values of elongation at rupture in 10,000 hr calculated by this means are given in Table V.

Reduction of area at rupture for long rupture times may be estimated by using the same technique as for elongation.

Fractures of steel D (5 Cr, $\frac{1}{2}$ Mo (Ti)) were elliptical, indicative of preferred orientation; the differences in the major and minor axes ranged from 0.003 to 0.036 in. In these instances, the average diameter was used in calculating the final area.

In contrast to the austenitic grades of steel, reported upon earlier (**3**), the ferritic grades show excellent ductility at rupture, even for the longest times, Table V. This is presumably associated with the relative freedom from intergranular fracture, compared to the austenitic grades (see subsequent discussion on mode of fracture).

TABLE VI.—ROOM TEMPERATURE TENSILE STRENGTH, IMPACT STRENGTH, AND HARDNESS BEFORE AND AFTER CREEP TEST.[b]

Steel[a]	Type	Creep Test,[b] deg Fahr	Diamond Pyramid Hardness	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	⅔ Size Charpy Notch Impact, ft-lb
				0.1 per cent Offset	0.2 per cent Offset				
B	5 Cr, ½ Mo(Si)	Before test	168	53 800[c]		87 900	31.0	65.4	29
B	5 Cr, ½ Mo(Si)	1250[d]	166	52 800[a]		82 000	32.5	70.0	28
B	5 Cr, ½ Mo(Si)	1500	154 (l.g.)[e] 164 (f.g.)[e]	47 300[c]		76 300	36.5	75.3	43
E	8 Cr, 1 Mo	Before test	142	...	31 200	75 000	31.0	66.1	23
E	8 Cr, 1 Mo	1100	139	...	...	...	...	...	26.5
E	8 Cr, 1 Mo	1300	131	26 800	27 100	67 800	35.5	69.6	25
F	8 Cr, 1 Mo(Cb)	Before test	143	...	34 800	71 400	33.5	74.9	41
F	8 Cr, 1 Mo(Cb)	1100	138	...	...	...	...	...	21
F	8 Cr, 1 Mo(Cb)	1300	132	30 000	30 500	66 900	37.0	76.1	41
G	12 Cr	Before test	249	88 900	92 800	112 500	21.0	73.3	37
G	12 Cr	1000	221	78 700	80 600	100 000	23.0	71.5	35
G	12 Cr	1200	158	53 500	54 200	81 600	30.0	75.3	44
H	12 Cr, ½ Mo	Before test	231	80 600	83 000	103 300	23.0	67.6	34
H	12 Cr, ½ Mo	1000	225	78 100	80 100	102 000	24.0	71.2	35
H	12 Cr, ½ Mo	1100	201	58 600	59 100	87 200	29.5	74.0	38
H	12 Cr, ½ Mo	1300	158	43 600	42 900	74 900	38.0	75.2	40

[a] Chemical composition and heat treatment in Table I.
[b] Creep tests of 3000 hr duration, unless otherwise noted.
[c] Yield point.
[d] Creep test of 1500 hr.
[e] l.g.—large grain area; f.g.—fine grain area.

Mechanical Properties at Room Temperature After Test:

Hardness, tension, and notch impact tests were made at room temperature on specimens machined from the standard creep specimens after 3000 hr tests. As creep tests were made for just five of the steels, B, E, F, G, and H, only these steels could be mechanically tested after creep.

The effect of time and temperature during creep on subsequent room-temperature properties, Table VI, was negligible for steel B, but steel F showed loss of notch impact strength after testing at 1100 F; the reason for this change was not apparent from examination of the microstructure. Steel B showed an increase in impact strength and slightly lower yield strength and tensile strength after the 1500 F tests. Steels G and H showed a lowering of yield strength, tensile strength, and hardness with increasing creep test temperature. Steel B tested at 1500 F exhibited a mixed grain size; the structure after test is discussed in greater detail in a later section of this report.

TABLE VII.—HARDNESS OF STEEL B (5 Cr, ½ Mo(Si)) AFTER CREEP-RUPTURE TESTS.[a]

Temperature, deg Fahr	Stress, psi	Rupture Time, hr	Diamond Pyramid Hardness, 20 kg Load		
			At Fracture	⅜ in. from Fracture	⅝ in. from Fracture
1000	40 000	0.25	250	218	......
	30 000	7.0	236	209	......
	20 000	192.0	254	205	......
	15 000	1603.0	233	186	......
1250	15 000	0.3	185	185	......
	10 000	3.3	176	177	......
	7 500	15.5	186	187	......
	5 000	162.0	145	172	......
	3 000	1466.0	101	154	......
1500	4 000	1.0	136[b]	143[c]	163[d]
	2 500	7.5	118[b]	151[c]	155[d]
	1 500	48.0	109[b]	142[c]	150[d]
	750	743.0	104[b]	142[c]	140[d]

[a] Initial hardness, 168 DPH.
[b] The large number of voids affects this value (Fig. 15 (*b*)).
[c] Coarse grain region (see Fig. 15).
[d] Fine grain region (see Fig. 15).

Hardness tests were made on metallographically prepared longitudinal sections of creep-rupture specimens of steel B after test in regions adjacent to the fracture and approximately $\frac{3}{8}$ in. from the fracture, with results listed in Table VII. Counteracting factors influencing hardness in these regions are plastic deformation (strain hardening) and recrystallization or grain growth; the formation of voids also has an effect. These conditions are illustrated in Figs. 15 and 16, where elongated grains in the "necked-down" region of a strain-hardened area are shown in Fig. 16(*a*), (*b*), and (*c*) and equiaxed grains as well as some voids are exhibited in Fig. 16(*d*), (*e*), and (*f*), and Fig. 15(*b*). As can be seen from Table VII, the hardness in sections with elongated grains (1000 F tests) was considerably greater than the initial hardness, whereas regions with equiaxed grains adjacent to fractures were lower in hardness (1500 F tests). However, it is to be remembered that the voids influence the measured hardness in these regions. This appreciable increase in hardness in the "necked-down" fracture regions with elongated grains is similar to earlier findings for plain carbon steels tested at 850 F (7).

STRUCTURAL STUDIES

All of the specimens which had been tested in creep and creep-rupture were examined for: (*a*) microstructural changes incurred during test and (*b*) evidence of the mechanism of plastic flow and fracture of the steels tested.

Initial Microstructure:

The microstructures of the eleven steels heat-treated in accordance with commercial practice, Table I, are shown in Fig. 14. Steel A (5 Cr, $\frac{1}{2}$ Mo) contains both lamellar carbide (pearlite) and spheroidized carbide, suggesting that the carbide did not completely dissolve during the austenitizing treatment. Steel B (5 Cr, $\frac{1}{2}$ Mo(Si)) shows a ferrite matrix and spheroidized carbides; the austenitizing temperature of 1600 F is probably below the lower critical temperature for this steel or at least not far above it. Steels C (5 Cr, $\frac{1}{2}$ Mo(Cb)) and D (5 Cr, $\frac{1}{2}$ Mo(Ti)) were not heated above their critical temperatures and exhibit ferrite matrices containing general precipitates of fine carbides and typical CbC, CbN and TiC, TiN particles, respectively.

Of the 8 per cent chromium steels, E (8 Cr, 1 Mo) exhibits a ferrite matrix with fine spheroidized carbides and poorly defined pearlite, whereas F (8 Cr, 1 Mo(Cb)) consists of a ferrite matrix with a general precipitate of fine carbides and typical CbC, CbN particles.

The 12 per cent chromium steels G (12 Cr), H (12 Cr, $\frac{1}{2}$ Mo), I (12 Cr, $\frac{1}{2}$ Mo(B)) and J (12 Cr, $\frac{1}{2}$ Mo(Cb)), are all substantially similar in structure; however, steel J had a finer grain size. Microstructurally these steels consisted of tempered martensite and delta ferrite; the austenitizing temperature of 1750 F evidently lies in the austenite-ferrite region for these steels.

Steel K (17 Cr), which did not form austenite during the heat treatment, shows a ferrite matrix with carbides within the grains and at grain boundaries; partially spheroidized pearlite was exhibited in many areas.

Microstructures After Creep and Creep-Rupture Tests:

The microstructure of steel A (5 Cr, $\frac{1}{2}$ Mo) remained substantially unchanged during tests at 900 and 1050 F; however; carbide spheroidization occurred in the specimens tested at 1200 F, the degree increasing with time of exposure. Recrystallization in the specimen tested at 1200 F for 1577 hr was shown by the presence in regions adjacent to the fracture of some equiaxed grains smaller than in the original steel.

Steel B (5 Cr, $\frac{1}{2}$ Mo(Si)) remained unchanged except for recrystallization in the fracture regions of specimens tested at 1250 and 1500 F. Specimens tested at 1250 F contained small equiaxed grains in the fracture region except that the specimen tested for the longest time, 1466 hr, also exhibited some large grains. At 1500 F, a range of grain sizes, illustrated in Fig. 15, was observed in the recrystallized zone. Examination of several specimens from tests at 1500 F under a common stress (3000 psi) which were interrupted at different fractions of the rupture time, the last of which had undergone an extension of 56 per cent (of a total of 141 per cent), failed to show evidence of recrystallization. One is thus forced to conclude that under these test conditions, at least, recrystallization does not begin until quite late in the test. It is even possible that, being dependent on time as well as degree of deformation, it does not begin until after fracture, although power to the test furnace is shut off automatically when the specimen breaks and cooling commences immediately. The coarseness of the carbides in these three specimens indicated coalescence during test. The specimen that ruptured in 723 hr at 1500 F showed relatively large equiaxed grains throughout the specimen with voids at grain boundaries, predominantly at the junctions of several grains, Fig. 16. Few carbides were present in this specimen, presumably as a result of decarburization. The 3000-hr specimen tested in creep at 1500 F showed a duplex grain size with some very large grains.

Steels C (5 Cr, $\frac{1}{2}$ Mo(Cb)) and D (5 Cr, $\frac{1}{2}$ Mo(Ti)) remained structurally the same throughout the creep-rupture tests at all temperatures except that steel C tested at 1200 F for 1000 hr showed a coarser general precipitate.

Steel E (8 Cr, 1 Mo) tested at 1100 F remained unchanged for rupture times up to 154 hr, but after 2542 hr the pearlite had partially spheroidized. The specimens tested up to 100 hr at 1300 F showed carbide spheroidization and partial recrystallization in regions adjacent to fracture, and the 6080-hr specimen exhibited fewer but very coarse carbides and equiaxed grains of the same size throughout the specimen.

During the tests at 1100 F, steel F (8 Cr, 1 Mo(Cb)) remained unchanged; however, the 1300 F specimens exhibited some recrystallization in the "necked-down" regions of the specimens which broke in times up to 40 hr, and the specimens which ruptured after longer test times remained equiaxed, Fig. 17. A heavy general precipitate, believed to be nitride, formed adjacent to intergranular cracks in the long time 1300 F specimens.

Steel G (12 Cr) tested at 1000 F remained substantially unchanged except for a fine general precipitation in the delta ferrite particles of the specimens tested for long times (3000 and 5358 hr). After tests at 1300 F, the steels consisted of alpha ferrite with resolvable carbides and also delta ferrite with a somewhat finer carbide precipitate; the carbides increased in size with longer times of test so that after the 2608 hr rupture time the delta regions were indistinguishable from the matrix.

Steel H (12 Cr, $\frac{1}{2}$ Mo) remained unchanged after the shorter test times at 1000 F, but after the 1457 and 3000-hr tests, the delta ferrite particles were darkened by a very fine general precipitate. During tests at 1100 F, no changes were found in the 1.25 and 5.5-hr specimens; however, for test times longer than 100 hr the matrix

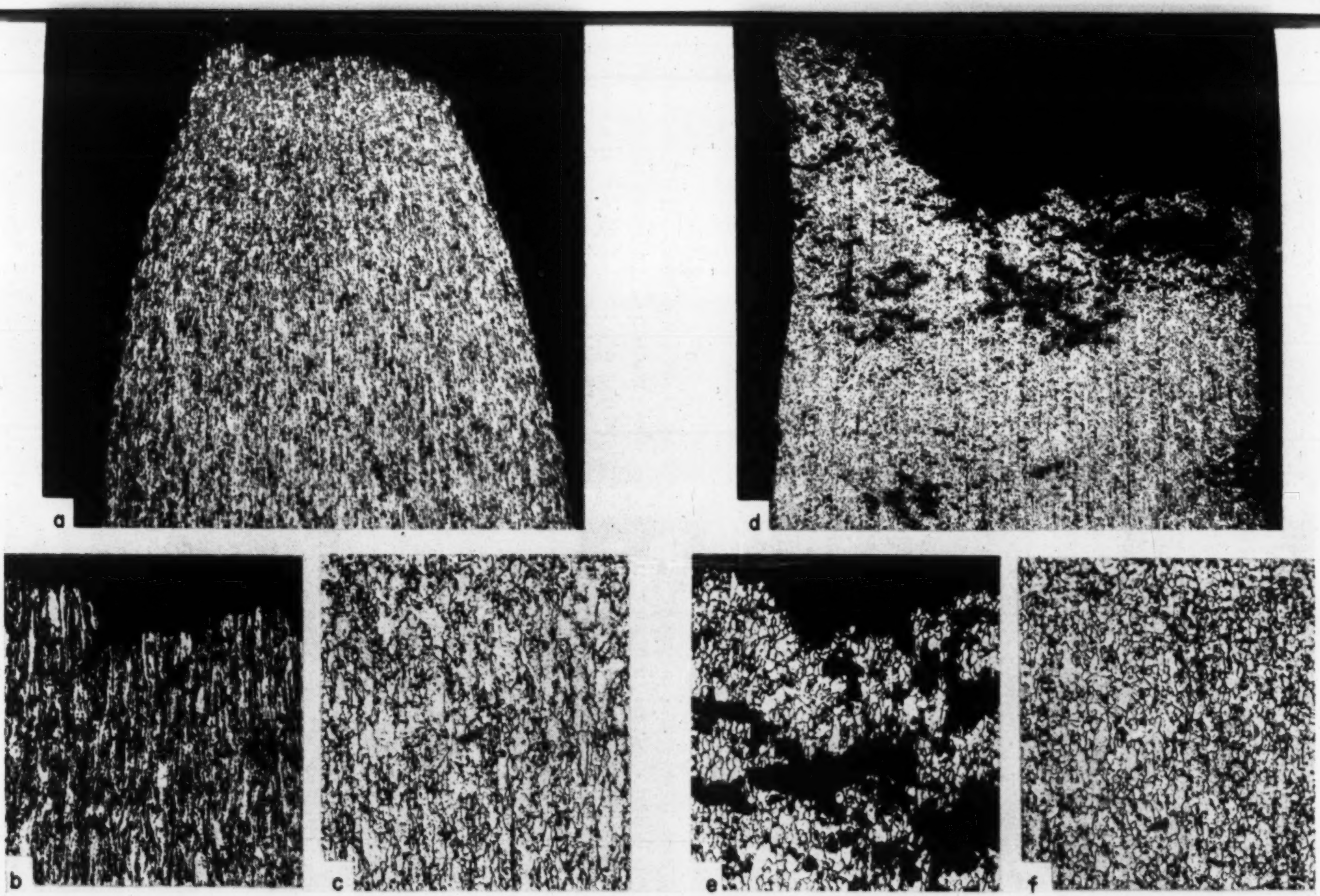

FIG. 17.—Longitudinal Sections of Creep-Rupture Specimens of Steel F (8 Cr, 1 Mo (Cb)) Tested at 1300 F.

(*a*), (*b*), (*c*) Specimen failed in 3.8 hr.
(*d*), (*e*), (*f*) Specimen failed in 1404 hr.
Magnifications of (*a*) and (*d*) are 25×; (*b*), (*c*), (*e*), and (*f*) are 100×. (Reduced to $^7/_{10}$ size in reproduction.)

consisted of ferrite and fine carbides, and the delta ferrite regions contained a fine general precipitate. The specimens tested at 1300 F exhibited a ferrite matrix with a general carbide precipitate and delta ferrite particles with a general precipitate; in the longer-time specimens (158 hr and longer) carbide coalescence was found and the delta regions were no longer distinguishable.

No microstructural changes were found in steels I (12 Cr, $\frac{1}{2}$ Mo(B)) and J (12 Cr, $\frac{1}{2}$ Mo(Cb)) after tests at 1000 F.

After tests at 900, 1050, and short rupture times (1.2 and 23.2 hr) at 1200 F, steel K (17 Cr) remained unchanged. Tests for 418 and 2871 hr at 1200 F, however, resulted in carbide growth and some evidence of recrystallization in the "necked-down" regions of the fractures. After shorter times of test at 1350 F, elongated and some recrystallized equiaxed grains were found adjacent to the fracture. With increased time for rupture (150 and 1620 hr), the grains adjacent to the fracture were predominantly equiaxed, while elongated grains existed farther from the fracture, indicating the dependence of recrystallization on the degree of deformation; pronounced carbide coalescence was shown by presence of fewer and much larger carbides.

Some Observations on the Mechanism of Flow and Fracture at Elevated Temperatures:

The phenomena of plastic deformation (flow) and subsequent fracture during creep-rupture testing are so interrelated that both have to be considered in any discussion of the mechanism by which metal flows and finally ruptures. Plastic deformation involves an intragranular flow (slip), and a grain boundary flow (slipless flow), and there may be an associated recovery and recrystallization, all depending on the metal, test temperature, and strain rate. Fracture may be: (1) transgranular, that is, through the grains, often with a characteristic "necking-down" at the fracture or (2) intergranular, rupture propagating through grain boundaries, characterized in many cases, but not in all, by low elongation and reduction of area. An oversimplified explanation of plastic flow by creep, which has been widely accepted, is that a strain hardening occurs in the first or decreasing rate of creep stage which is followed by the second or constant creep rate stage characterized by a balance between strain hardening and annealing; the third or accelerating rate stage is caused by predominance of the annealing effect. If this general explanation, which does not take into consideration the structural variations which result from test conditions, were accurate, a softening should take place during third stage creep. Hardness tests in the fractured or "necked-down" regions of creep rupture specimens which deformed by third stage creep and in the matrix behind the "necked-down" region, Table VII, show that hardness may be actually greater in regions that had been subjected to accelerating creep than in the rest of the specimen or original material (see also (7)).

Typical types of flow and fracture are shown macroscopically in Fig. 18 and microscopically in Figs. 15 to 17. In Fig. 18(*a*) is shown a specimen after a characteristic intergranular fracture; the microstructure of this specimen, Fig. 17(*d*), (*e*), and (*f*), shows equiaxed grains, transverse intergranular voids and an irregular fracture surface. A characteristic transgranular fracture, for the same steel, with a greater elongation and reduction of area, is shown in Fig. 18(*b*); microstructurally this specimen, Fig. 17(*a*), (*b*), and (*c*), shows elongated grains, a few longitudinal voids and a

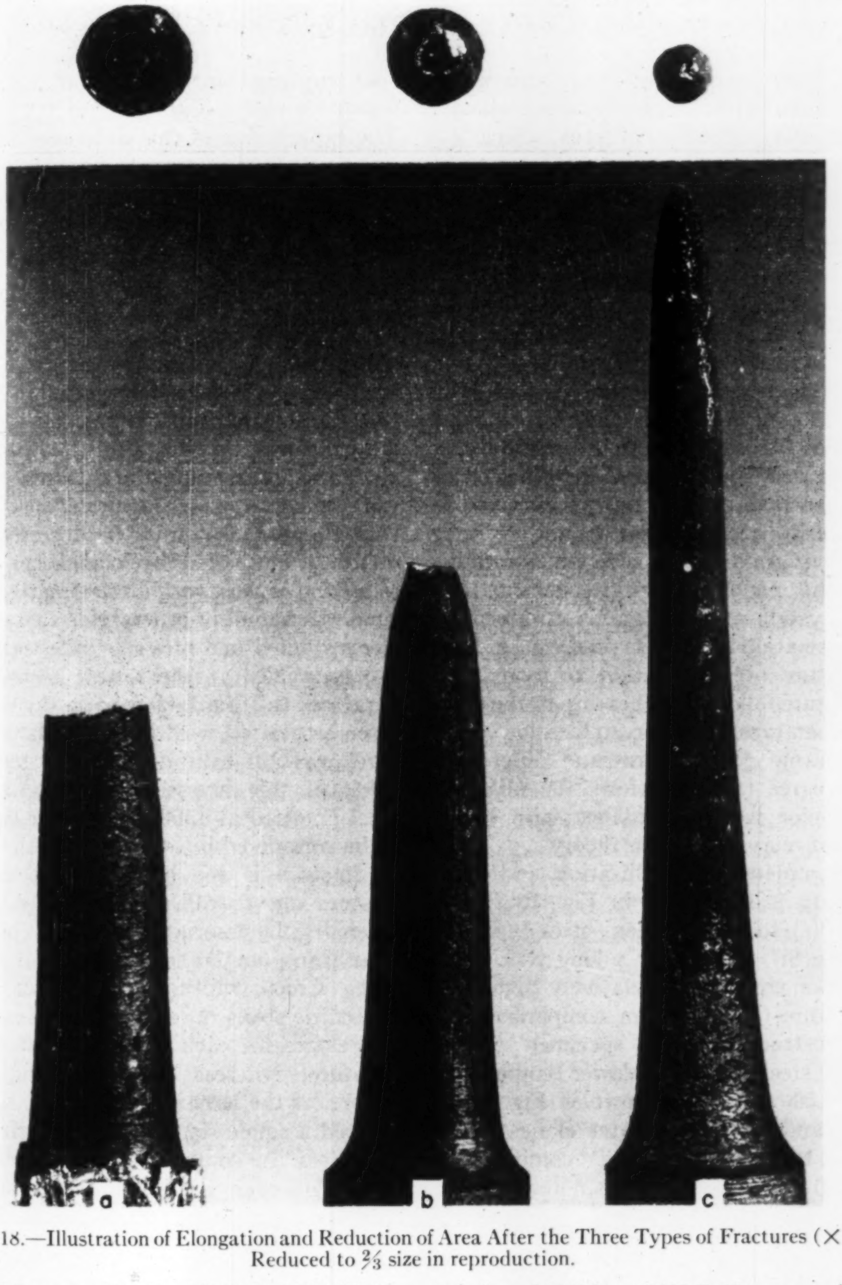

FIG. 18.—Illustration of Elongation and Reduction of Area After the Three Types of Fractures (× 4). Reduced to ⅔ size in reproduction.

(*a*) Intergranular, Steel F (8 Cr, 1 Mo (Cb)), rupture time of 1400 hr at 1300 F, 49 per cent elongation and 62 per cent R. A.

(*b*) Transgranular, Steel F (8 Cr, 1 Mo (Cb)), rupture time of 3.8 hr at 1300 F, 87 per cent elongation and 92 per cent R. A.

(*c*) Recrystallization, Steel B (5 Cr, 0.5 Mo (Si)), rupture time of 48 hr at 1500 F, 198 per cent elongation and 98.5 per cent R. A.

relatively smooth fracture surface. A specimen with unusually great elongation and reduction of area which exhibited recrystallization in the region of fracture is shown in Fig. 18(*c*); the microstructure of the fracture region, Fig. 15(*a*) to (*d*), shows a clear delineation between the recrystallized and unrecrystallized portions. A gradient of grain size in the recrystallized region is shown in the photomicrographs, ranging from fine at the fracture to coarse at junction of the matrix and recrystallized regions. It is evident from these illustrations that the degree of deformation at the temperature of test is effective in initiating recrystallization and forming various grain sizes, coarse grains forming in the region of critical deformation. Recrystallization diagrams obtained by deformation at the recrystallization temperature or deformation at room temperature followed by heating to the same temperature of recrystallization have the same general form and differ only in degree (10); therefore, the observed behavior is in accordance with established recrystallization theory.

Complete recrystallization and grain growth is illustrated in Fig. 16(*d*), (*e*), and (*f*) for a specimen, steel B (5 Cr, $\frac{1}{2}$ Mo(Si)), tested for a long time (slow rate of creep) at a relatively high temperature (1500 F). For comparison, the microstructure of a specimen of the same steel tested at a lower temperature for a short time is shown in Fig. 16(*a*), (*b*), and (*c*). The greater elongation in Fig. 16(*d*) (90 per cent) compared to Fig. 16(*a*) (51 per cent) is noteworthy.

Steel D (5 Cr, $\frac{1}{2}$ Mo(Ti)) exhibited pronounced elliptical fractures after tests at all temperatures owing presumably to preferred orientation in the material as received and heat treated. All of the ruptured specimens had a difference in major and minor axes of 0.003 to 0.009 in., with the exception of the specimen that ruptured in 3616 hr at 1200 F which showed a difference of 0.036 in. The explanation of this difference is not known. It is of interest to note that this steel initially exhibited a duplex grain size, whereas all of the other steels were fine grained (Table I).

Flow and Fracture of Austenitic versus Ferritic Steels:

Whereas, in general, austenitic steels are characterized by intergranular fracture for longer rupture times at elevated temperatures, ferritic steels, which include the group studied in this investigation, show a marked prevalence for transgranular fracture. A decrease in ductility, measured by elongation and reduction of area, with increasing rupture time was found, in general, for austenitic steels studied in a previous investigation (3), especially at the lowest test temperature; this marked drop in ductility, often associated with intergranular fracture, was not exhibited for the ferritic steels of this investigation, except for steel E tested at 1300 F. Another factor to be considered in connection with type of fracture is the break in the creep rupture curve which is generally considered to be associated with the change from transgranular to intergranular fracture. Creep rupture curves for the austenitic steels (3) exhibited a break or two slopes for each steel and test temperature, whereas the creep rupture curves for the ferritic steels of this study showed a single slope in the majority of the cases. In contrast to the austenitic steels, however, the break in the curve, when observed, did not always correspond with change of fracture type.

From the observations on fractures in austenitic and ferritic steels, it appears that austenitic steels offer a greater relative resistance than ferritic steels to intragranular flow and fracture at elevated temperatures so that the factors of the so-called "slipless" flow and grain boundary strength assume an important rôle in determining strength in creep and creep rupture. The greater intragranular strength of austenitic steels, which is presumably associated with the face-centered cubic lattice structure, is manifested in the higher creep and creep rupture strengths of austenitic steels compared to ferritic steels. If this reasoning is correct, it may also be predicted that whereas increased grain size may result in greater strength of austenitic steels, this should not be expected in ferritic steels within the temperature range investigated.

SUMMARY

Comparison of creep and creep-rupture strength of the three groups of steels (5 Cr, $\frac{1}{2}$ Mo, 8 Cr, 1 Mo, and 12 Cr, $\frac{1}{2}$ Mo) with and without additional alloying elements indicates that the steels containing columbium are strongest up to 1200 F; at higher temperatures the steels without any additional alloying elements other than molybdenum are slightly stronger. The 5 Cr, $\frac{1}{2}$ Mo steels with silicon or titanium are weaker at all test temperatures than the plain 5 Cr, $\frac{1}{2}$ Mo or 5 Cr, $\frac{1}{2}$ Mo(Cb) steels. Columbium addition to 8 Cr, 1 Mo steel increases strength up to 1200 F, but at higher temperatures the strength decreases. Plain 12 Cr steels are considerably weaker than 12 Cr, $\frac{1}{2}$ Mo steels with or without other alloying elements. Of the 12 Cr, $\frac{1}{2}$ Mo group tested at 1000 F, the 12 Cr, $\frac{1}{2}$ Mo(Cb) steel has the highest strength; the 12 Cr, $\frac{1}{2}$Mo(B) and 12 Cr, $\frac{1}{2}$ Mo steels are substantially equal in creep-rupture strength, but the boron-containing steel has higher creep strength. The 17 Cr steel is comparable in strength to the 5 Cr, $\frac{1}{2}$ Mo steel, which is at the lower strength level of the series of steels investigated.

Microstructural examination before and after creep and creep-rupture tests showed carbide spheroidization and recrystallization to be the most significant changes occurring during tests. No sigma was detected in steel K (17 Cr) by microscope or X-ray tests. Recrystallization was observed in several of the test specimens examined after test.

Acknowledgment:

The authors gratefully acknowledge the help of E. G. Houston in carrying out the creep and rupture tests and of W. B. Seens and W. L. Jensen in making short-time tension and notch-impact tests.

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DISCUSSION

MR. S. F. REITER[1] (*presented in written form*).—The authors have made another fine contribution to the information concerning the behavior of ferritic steels during high-temperature service. One of their conclusions, however, warrants qualification.

The authors hold that both titanium and silicon additions to 5 per cent chromium, ½ per cent molybdenum steels caused a considerable loss of high-temperature strength. In the case of silicon, their data are definitely confirmatory. However, steel D, containing 0.55 per cent titanium was not directly comparable with steel A since it (*a*) contained only 0.05 per cent carbon, and (*b*) received a low-temperature heat treatment at 1375 F, whereas steel A contained 0.13 per cent carbon and was heated to 1550 F. Certainly the microstructures of steels A and D as shown in Fig. 14 exhibit a great difference of structures. The low density of carbide particles in steel D is probably responsible for the coarse grain structure observed following the experimental heat treatment. It is quite probable that a 5 per cent chromium, ½ per cent molybdenum steel containing 0.55 per cent titanium might have equivalent or superior high-temperature properties to a titanium-free steel having the same carbon content, if both received the same higher temperature treatment.

MR. GEORGE F. COMSTOCK[2] (*presented in written form*).—This interesting and comprehensive paper will undoubtedly be referred to in the future as a standard reference to the subject, and it is unfortunate, therefore, that the limitations very frankly admitted by the authors near the top of the second column of p. 647 are not reflected to even the slightest degree in the summary. Few will notice the clarifying restrictions noted on p. 647, but many will learn from the summary that "the steels containing columbium are strongest," while the 5 per cent chromium, ½ per cent molybdenum steels with titanium are weaker than the plain 5 per cent chromium, ½ per cent molybdenum. It should be stated right here that this conclusion is drawn from only one steel of each type, heat treated in only one way.

When the analyses of the 5 per cent chromium steels containing columbium or titanium are examined, it is seen that the columbium:carbon ratio is 10, while the titanium:carbon ratio is 11. The former is about what is required to stabilize the carbon with columbium, so that this steel is properly representative of its class, but the latter is more than twice as high as is required for carbon stabilization by titanium. It has been shown in a previous publication that such a high titanium:carbon ratio impairs the high-temperature strength of 5 per cent chromium steel, and is not necessary to prevent air-hardening. Therefore, the authors' choice of such a steel for the extensive work done in preparing their paper seem peculiarly unfortunate, and largely nullifies their conclusions as applied to titanium 5 per cent chromium steels in general. The coarse grains that they noted in the microstructure would probably not have been found had the composition been properly representative of this grade of steel, and the rupture strength would certainly have been higher.

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[2] Assistant Director of Research, National Lead Co., Niagara Falls, N. Y.

The heat treatment of the authors' 5 per cent chromium-molybdenum-titanium steel might also be questioned. Heat treatment at 1375 F is good for this grade of steel, if it follows rolling or rapid cooling from a higher temperature such as 1600 or 1700 F. But if the steel had been annealed before the authors obtained it, the treatment at 1375 F would not correct the weakening effect of slow cooling from a high temperature. Thus the structure and properties may have been affected by such a treatment. Furnace cooling from 1375 F was also unfortunate, since air cooling of this grade is fully permissible, and may be better.

MR. R. A. LULA.[3]—What is the influence of delta ferrite on the creep strength of straight type 410 steel and 410 alloyed with columbium, molybdenum, and titanium? Second, is a completely martensitic structure necessary in order to obtain good creep strength?

MESSRS. E. J. DULIS AND G. V. SMITH (*authors' closure*).—In carrying out these tests, we fully recognized that neither the composition of a specific grade nor its heat treatment would necessarily be those for optimum strength at elevated temperatures. In fact, there is so little known about the effects of such variables on most of these grades that it seems highly unlikely that optimum conditions were realized. We called attention to this in the text of the paper, but, by an oversight, failed to again mention it in the summary.

It is therefore quite possible that the 5 per cent chromium, molybdenum, titanium steel, about which Messrs. Reiter and Comstock comment, has not shown in as favorable a light as it might otherwise have. By the same token, however, it is also possible that the columbium-containing grade, for example, might also be greatly improved by appropriate selection of compositon and heat treatment.

Whatever strength the 5 chromium-molybdenum, titanium grade might have under optimum conditions, it does appear that the strengths evaluated in our tests for this grade are reasonably representative of commercial practice. When comparison is made with the results of a survey[4] of data on these and other grades made by the Data and Publications Panel of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals, it develops that the specific values for rupture in 1000 hr for 5 chromium, molybdenum, titanium steel reported in the present paper fall slightly above an average curve drawn through the variation with temperature of the compiled data. Similar comparisons for the 5 Cr-Mo and 5 Cr-Mo-Si grades show the results of the present paper to fall slightly below the curves for the compiled data. No compiled data for 5 Cr-Mo-Cb are available.

We are unable to answer Mr. Lula's questions concerning the influence of delta ferrite in type 410 steels and the need for a fully martensitic microstructure.

Much work needs to be done to determine the conditions for optimum strength in these grades.

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[3] Research Metallurgist, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

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INFLUENCE OF SHARP NOTCHES ON THE STRESS-RUPTURE CHARACTERISTICS OF HEAT-RESISTING ALLOYS: PART II*

By W. F. Brown, Jr.,[1] M. H. Jones,[1] and D. P. Newman[1]

Synopsis

Sharply notched and smooth bar stress-rupture tests were carried out on a number of ferritic low-alloy steels, ferritic stainless steels, and austenitic alloys. The temperatures of testing in most cases covered the range of service application for the particular material. Data at these temperatures are reported for rupture times between 0.1 and 1000 hr.

The general time-temperature dependence of the notch effect in stress-rupture tests has been confirmed for a wide variety of materials. An analysis of the ductility data reveals that the smooth-bar reduction of area at fracture yields no definite information regarding the notch rupture sensitivity of a given alloy. The notch ductilities were found to conform with the previous hypothesis that the weakening effect of a sharp notch is associated with a retained stress concentration.

Consideration of published data for total creep on certain of the alloys indicates that in some cases design for a certain total deformation would be limited by the notch strength.

In a previous publication (1)[2] the authors described some results of an investigation into the notch-rupture sensitivity of several heat-resistant alloys of current interest to the gas turbine designer. Data for the following alloys were reported: (1) low-alloy steels, "17-22A" (S) and SAE 4340, (2) ferritic stainless steels, Crucible 422 and AMS 5616, and (3) austenitic alloys, Haynes 88 and Inconel X. The notch specimens employed possessed an extremely sharp, 50 per cent, 60-deg V notch. With the exception of "17-22A" (S) and Inconel X the data were available at only one or at the most two test temperatures.

This paper reports the results of an extension of the previous investigation to include the alloys "17-22A" (V), AMS 5602, and Nimonic 80A. Additional tests have also been made on the previously reported alloys to extend the test temperature range to cover that normally encountered in service applications.

On the basis of the previously reported data it appeared possible to draw several tentative conclusions: (1) The weakening effect of a severe notch is probably a universal phenomenon common in varying degree to all creep-resistant alloys; (2) There is no definite limiting value of the smooth-bar ductility, above which freedom from notch sensitivity is assured; (3) Notch sensitivity is associated with values of notch ductility less than

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Research Metallurgists, Lewis Flight Propulsion Laboratory, National Advisory Committee for Aeronautics, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 676.

3 per cent, and (4) Notch-rupture sensitivity is a time-temperature dependent phenomenon. The rather extensive data now available permit careful re-examination of these conclusions. In addition, an attempt has been made to review the published notch-rupture strength data to indicate the effects of alloy composition.

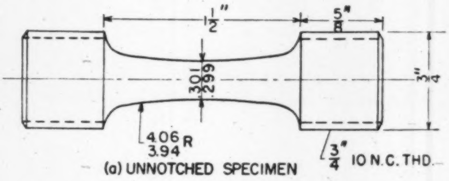
TABLE I.—COMPOSITION, HEAT TREATMENT, AND HARDNESS OF ALLOYS TESTED.

Alloy	Composition, per cent								Heat Treatment	Rockwell Hardness, C Scale
	Carbon	Chromium	Nickel	Molybdenum	Manganese	Silicon	Iron	Others		
"17-22A" (S)	0.30	1.25	0.25	0.52	0.63	0.60	Balance	V—0.25	1725 F, ½ hr, Air: 1200 F, 6 hr, Air	33
SAE 4340	0.37	0.74	1.75	0.23	0.60	0.21	Balance	....	1750 F, ½ hr, Air: 1200 F, 2 hr, Air	29
"17-22A" (V)	0.26	1.34	0.34	0.51	0.62	0.68	Balance	V—0.77	1800 F, ½ hr, Air: 1200 F, 6 hr, Air	35
AMS-5602	0.16	4.94	....	0.59	0.41	0.40	Balance	....	1725 F, 1 hr, Air: 1200 F, 6 hr, Air	20
AMS-5616	0.12	12.88	1.98	0.50	0.35	0.31	Balance	W—2.86	1800 F, 2 hr, Air: 1050 F, 2 hr, Air	36
Crucible 422	0.23	13.19	0.65	1.03	0.81	0.16	Balance	V—0.25 W—0.84	1900 F, ½ hr, Oil: 1200 F, 2 hr, Air	33
Haynes 88	0.07	12.48	15.48	2.74	1.38	0.51	Balance	W—0.77 Ti—0.45 B—0.11	2100 F, 1 hr, Air: Worked 40 per cent at 1400 F	28
Nimonic 80A[a]	0.1 max	20	Balance	....	1.0 max	1.0 max	5.0 max	Ti—2.2 Al—1.1 Co—2.0	1976 F, 8 hr, Water: 1292 F, 16 hr, Air	27
Inconel X[a]	0.08	15	72	....	0.70	0.50	7	Ti—2.5 Cb—1.0 Al—0.7	2100 F, 4 hr, Oil: 1550 F, 24 hr, Air: 1300 F, 20 hr, Air	32

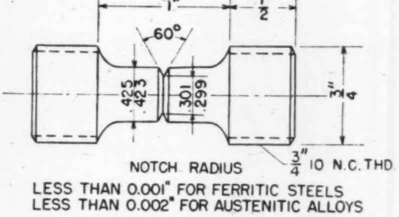
[a] Nominal composition.

MATERIAL AND PROCEDURE

The composition, heat treatment, and the hardness of the alloys tested are shown in Table I. With the exception of Haynes 88 and AMS 5602 all material was received as $\frac{3}{4}$- or 1-in. hot-rolled bar stock. The Haynes 88 was furnished as $\frac{11}{16}$-in. diameter bars in the finished rolled condition as shown in Table I. The AMS 5602 specimens were taken from 12-in. diameter, 1-in. thick disks. These disks were forged from 5-in. diameter, 10-in. cylindrical billets which were finished at 1850 F. After forging, the disks were annealed at 1575 F for 8 hr and then given the heat treatment shown in Table I. Specimens were cut from blanks in both radial and tangential positions near the rim.



(a) UNNOTCHED SPECIMEN


1"
1/2"
60°
.425
.423
.301
.299
3/4"
3/4" 10 N.C. THD
NOTCH RADIUS
LESS THAN 0.001" FOR FERRITIC STEELS
LESS THAN 0.002" FOR AUSTENITIC ALLOYS

(b) NOTCHED SPECIMEN

FIG. 1.—Stress-Rupture Specimens.

The specimen types are shown in Fig.

1. The large radius was used on the smooth bars to insure that fracture would occur in the minimum section. The notched bars possessed notch radii less than 0.001 in. for all alloys except where otherwise noted. This radius provides a notch sharpness (ratio of one half the diameter at the minimum section to the radius at the notch bottom) of approximately 150 which corresponds to a technical elastic stress concentration factor of approximately five (2).[3]

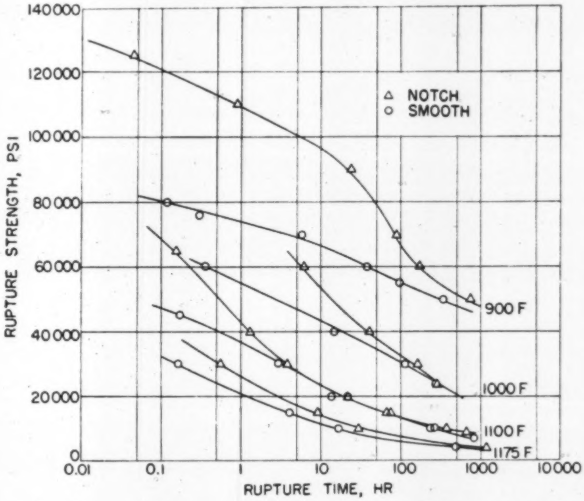


FIG. 2.—Notch and Smooth Rupture Strength for SAE 4340 at Several Temperatures.

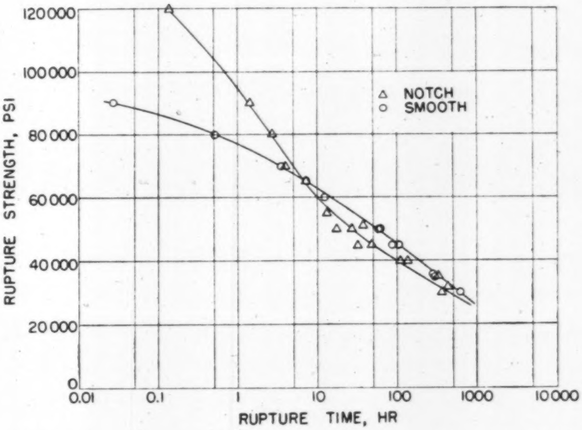


FIG. 3.—Notch and Smooth Rupture Strength for "17-22A" (V) at 1100 F.

The testing technique and equipment were previously described in detail (1), and with the exceptions discussed below were not changed.

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[3] According to Neuber (**2**), the technical stress concentration factor has a limiting value for extremely sharp notches. This limit results from the assumption that the material is composed of finite particles. The size of these particles and consequently the limiting value may be different for different materials.

The first portion of this investigation was carried out using new creep machines and with new and carefully machined loading rods. Under these conditions the notch strength data showed in most cases a reproducibility as good as that obtained with the smooth bars, indicating that eccentricity effects if present were either not large or nearly constant from test to test. As the investigation expanded, various types of older creep machines and loading rods were used. Under such conditions it was found that high and extremely variable eccentricities were produced which did cause excessive scatter in the notch properties of the brittle alloys. It was found that the eccentricity could be reduced greatly by

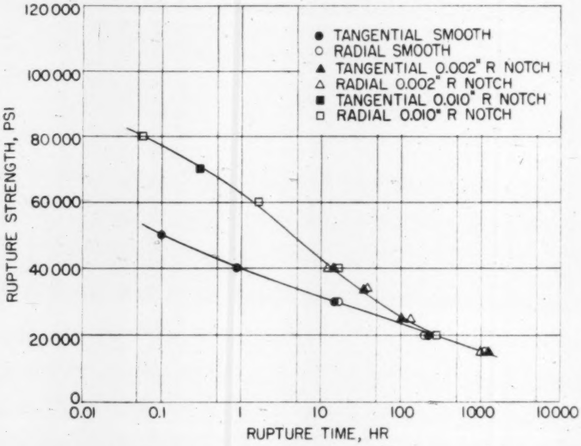

Fig. 4.—Notch and Smooth Rupture Strength for AMS 5602 at 1100 F (Specimens Cut from a Disk).

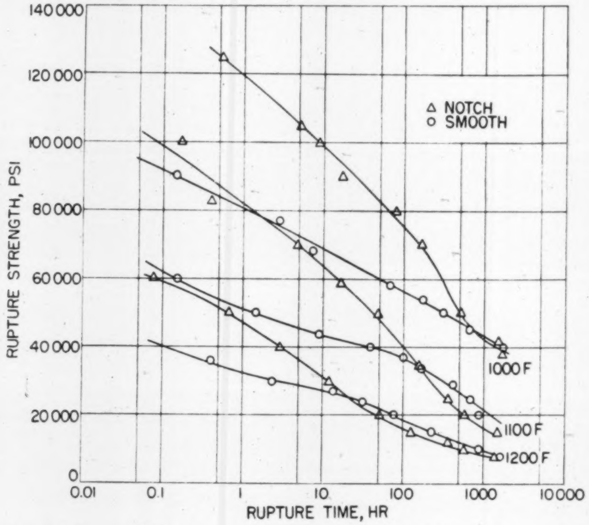

Fig. 5. Notch and Smooth Rupture Strength for AMS 5616 at Several Temperatures.

two methods: (1) careful machining of both specimens and loading rods to reduce the eccentricities of these parts to a minimum when assembled, and (2) the introduction of universal joints between the loading rods and the heads of the creep machines.

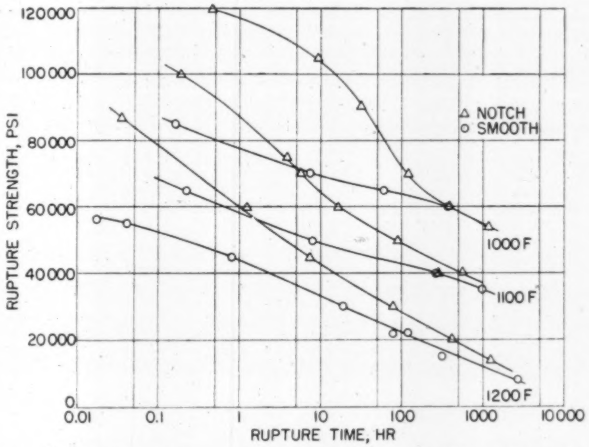

FIG. 6.—Notch and Smooth Rupture Strength for Crucible 422 at Several Temperatures.

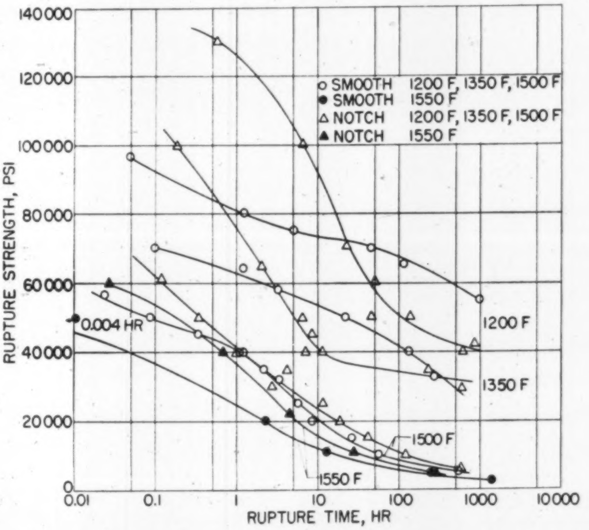

FIG. 7.—Notch and Smooth Rupture Strength for Haynes 88 at Several Temperatures.

RESULTS AND DISCUSSION

The notch and smooth bar rupture strength data for the various alloys are assembled in Figs. 2 to 8. Smooth and notched bar ductilities are shown in Figs. 9 to 15. The ratio of the notch strength to the smooth-bar strength, designated as the "notch rupture-strength ratio" has been added to the latter figures.

Rupture Strength of Ferritic Alloys:

The influence of a notch on the rupture properties of the ferritic steels, SAE 4340, "17-22A" (V), AMS 5602, AMS 5616, and Crucible 422 is fundamentally similar and identical to that reported previously for the alloy "17-22A" (S). The magnitude of notch weakening and its time range vary both with the alloy and with the test temperature. In general, the results confirm the conclusions that the notch sensitivity is time-temperature dependent in that the time of onset of notch sensitivity and the maximum notch weakening decrease with increasing temperature. At sufficiently high temperatures or long times at a given temperature, it is possible to "recover" the notch strength as shown in Fig. 2 for SAE 4340, and Fig. 7 for Haynes 88.

The low-alloy high-strength ferritic steel "17-22A" (V), Fig. 3, exhibits the greatest notch sensitivity (at 1100 F) in the time range investigated. This pronounced effect for a similar steel "17-22A" (S) was previously reported (1). These steels are characterized by a rapid decrease of notch strength in a restricted

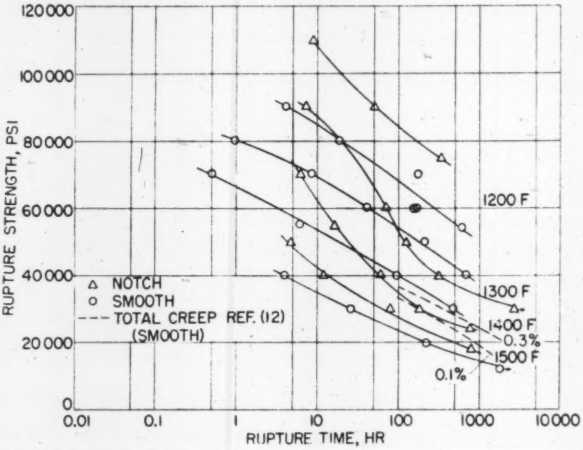

FIG. 8.—Notch and Smooth Rupture Strength for Nimonic 80A at Several Temperatures.

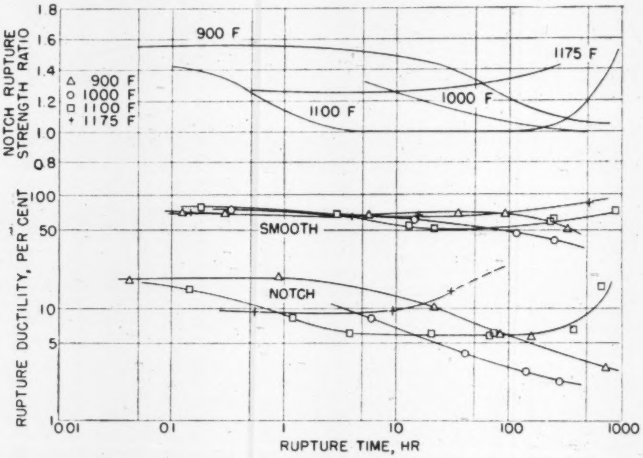

FIG. 9.—Notch Rupture Strength Ratio and Rupture Ductilities for SAE 4340 at Several Temperatures.

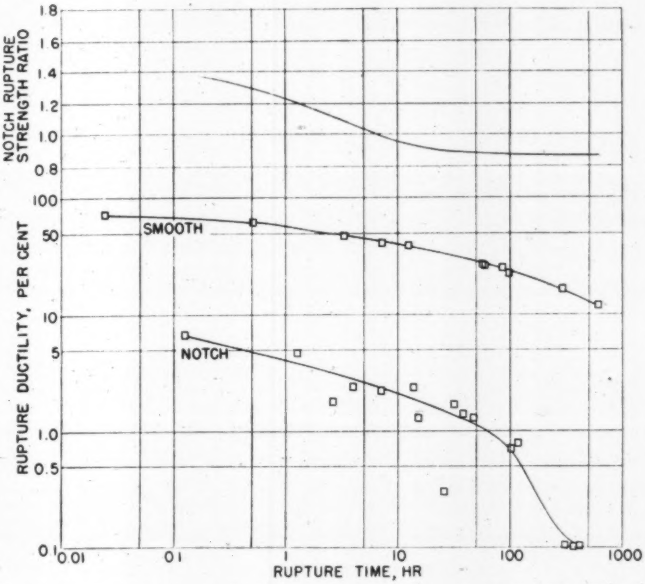

FIG. 10.—Notch Rupture Strength Ratio and Rupture Ductilities for "17-22A" (V) at 1100 F.

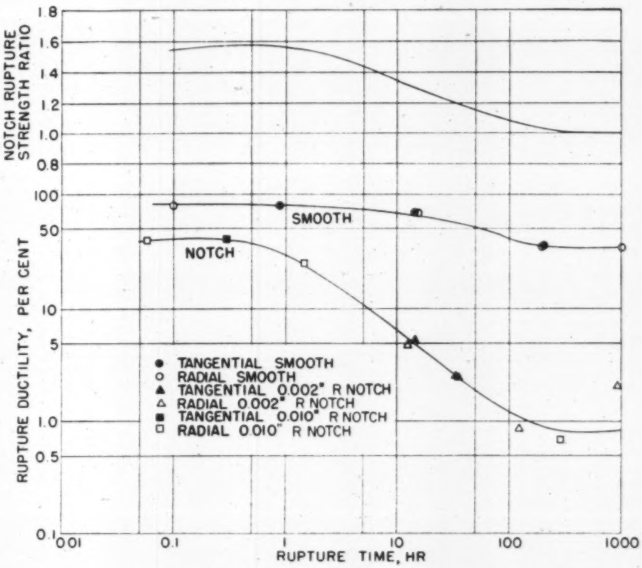

FIG. 11.—Notch Rupture Strength Ratio and Rupture Ductilities for AMS 5602 at 1100 F.

range of rupture times. This rapid decrease, which occurs at comparatively short time, combined with a rather stable smooth-bar strength results in high notch sensitivities at short rupture times. In contrast, the higher chromium steels, AMS 5602, AMS 5616, Crucible 422, Figs. 4, 5, 6, as well as the low-strength steel SAE 4340 exhibit a more gradual decrease of notch strength with increasing time to rupture. Notch weakening was observed only for the AMS 5616 and occurred at considerably higher times than in the lower alloyed steels. Both SAE 4340 and Crucible 422 are free from notch weakening for the ranges of times and temperatures investigated. For either of these materials the highest test temperatures resulted in a complete disappearance of the notch sensitivity. From

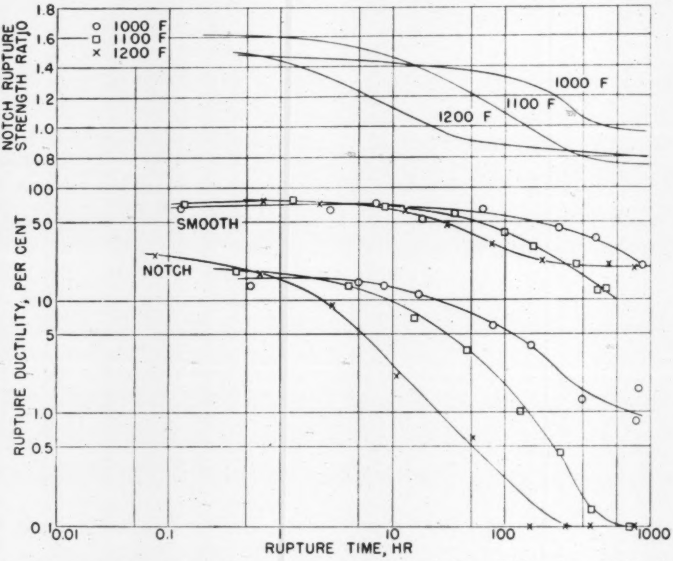

FIG. 12.—Notch Rupture Strength Ratio and Rupture Ductilities for AMS 5616 at Several Temperatures.

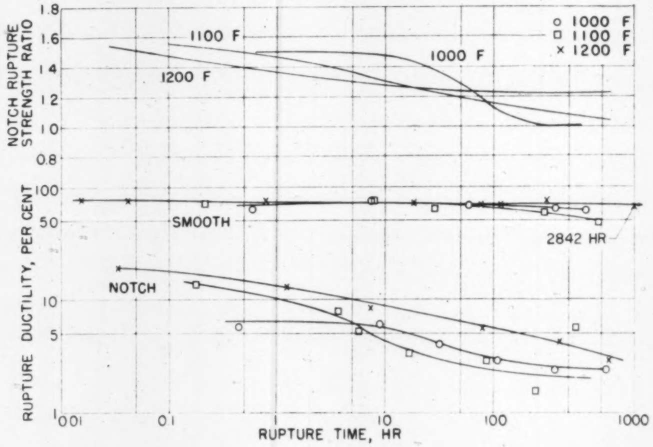

FIG. 13.—Notch Rupture Strength Ratio and Rupture Ductilities for Crucible 422 at Several Temperatures.

the trend of the curves it is not likely that notch weakening would be encountered at any time to rupture for the temperatures investigated, or at higher temperatures.

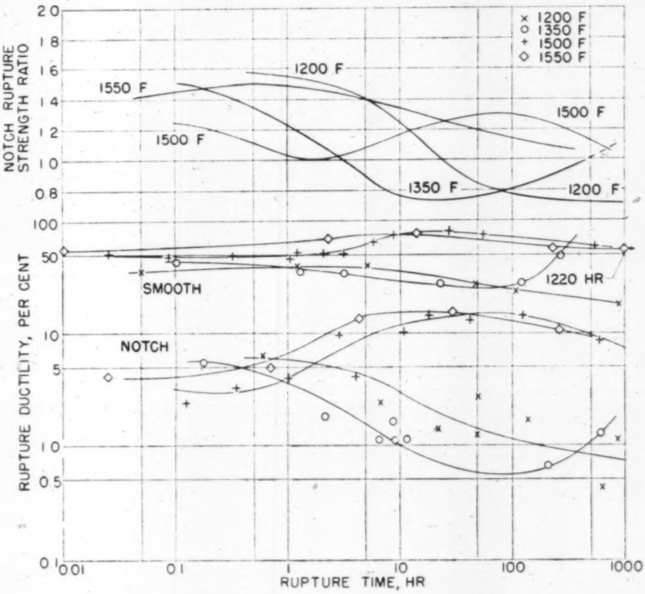

Fig. 14.—Notch Rupture Strength Ratio and Rupture Ductilities for Haynes 88 at Several Temperatures.

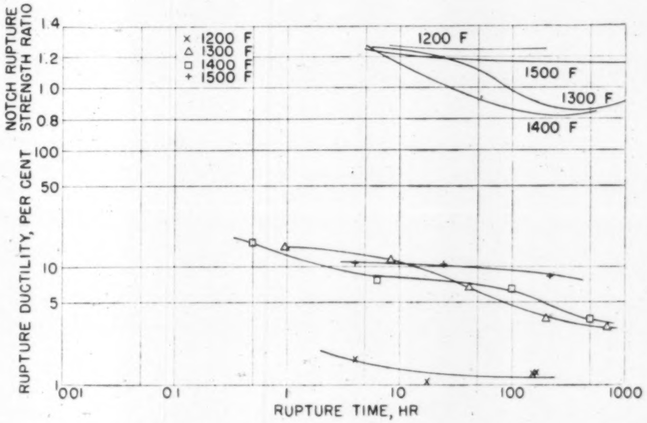

Fig. 15.—Notch Rupture Strength Ratio and Smooth Rupture Ductility for Nimonic 80A at Several Temperatures.

It should be noted that the tests on AMS 5602, Fig. 4, were made at notch radii of 0.002 and 0.010 in. These correspond to notch sharpnesses of 75 and 15, respectively, as compared with 150 used for the other materials in this group. In addition, the effect of directionality in a disk forging is shown, specimens having been taken both radially and tangentially near the rim. Within the limits of scattering, the large variation in notch sharpness had no effect. This is expected for a ductile material (**3**), and there is no reason to believe a sharper notch would weaken the alloy. There was practically no difference between the notch or smooth bar strengths for radial and tangential specimens.

Rupture Strength of Austenitic Alloys:

The rupture properties of the austenitic alloys Haynes 88 and Nimonic 80A are shown in Figs. 7 and 8. Both these alloys exhibit a response to notching very similar to that previously described for the low-alloy steels. However, the notch sensitivity is shifted to higher temperatures. For Haynes 88 the previously mentioned "recovery" effect is quite pronounced at 1350 F and complete recovery has occurred at even the shortest rupture times at 1500 F.

The behavior of these two alloys is different from that previously reported for Inconel X (1) in the same range of temperatures in that there is a distinct decrease in the notch strength at a given rupture time with an increase in the testing temperature, whereas for Inconel X the notch rupture strength between 1 and 1000 hr was practically independent of temperature for test temperatures from 1350 to 1500 F.

Rupture Ductilities:

Both smooth and notch bar ductilities are shown in Figs. 9 to 15 for all alloys except Nimonic 80A. The notch ductility of this alloy, like Inconel X, was impossible to measure because of the extremely irregular nature of the fracture. The notch rupture - strength ratio has been added to the representations to show the correlation between the notch effect and the measured ductilities. For each alloy there is a close correspondence between the trends of the notch ductility and the notch rupture - strength ratio.[4] No such general correspondence is observed for the smooth-bar ductilities. Thus, the smooth-bar ductility in the case of AMS 5616, Fig. 12, remains constant at short time to rupture, while the notch ductility and notch rupture - strength ratio are decreasing. Further-

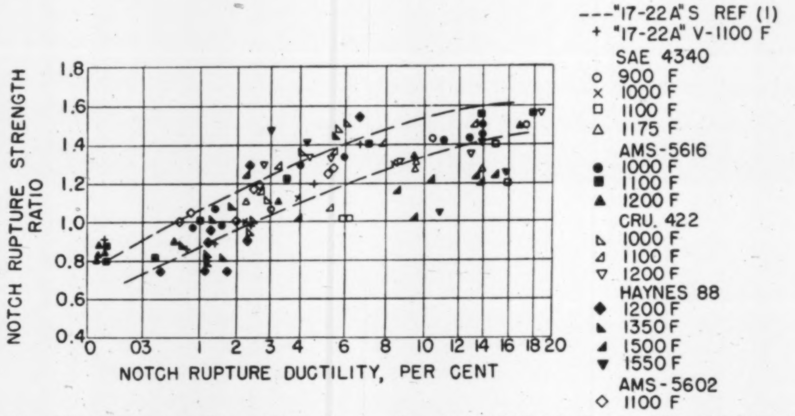

FIG. 16.—Notch Rupture Strength Ratio as a Function of Notch Rupture Ductility for Several Alloys at the Indicated Temperatures.

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[4] The relationships between notch rupture strength ratio and notch ductility for Haynes 88 at 1500 and 1550 F are complicated by recrystallization which was first observed to occur for rupture times of approximately 2 hr at 1500 F.

more, the smooth-bar ductility of Crucible 422, Fig. 13, changes only slightly with increasing rupture time at any of the test temperatures, while pronounced variations are noted in the notch rupture - strength ratio and notch ductility.

If notch weakening is related to the presence of a retained stress concentration at fracture, then the notch ductility should be extremely low for notch rupture - strength ratios less than unity. In a previous publication (1) it was pointed out that for "17-22A" (S), tested at 900, 1000, 1100, and 1200 F, the notch rupture - strength ratio decreased continuously with decreasing notch ductility and that notch weakening was always associated with values of the notch ductility less than 2 to 3 per cent. The notch rupture - strength ratio for the alloys reported in this paper has been plotted against the notch ductility in Fig. 16. Because of wide differences in the deformation characteristics among the alloys investigated and because of the influence of temperatures on these characteristics, no universal relation is expected, such as that reported by Brown and Sachs (3) for tension tests on low-alloy steels. However, without exception notch strength ratios less than unity are associated with notch ductilities less than approximately 2.5 per cent.

It is evident from the curves in Figs. 9 to 15 that there is no limiting value of smooth-bar ductility corresponding to the onset of notch sensitivity, nor is an alloy possessing a very high smooth-bar ductility necessarily free from notch weakening. Thus, for AMS 5616, Fig. 12, the value of the smooth-bar ductility is approximately 55 per cent for a notch strength ratio of unity (30 hr at 1200 F); while for Haynes 88, Fig. 14, at the same temperature (22 hr) a smooth-bar ductility of 30 per cent corresponds to a notch strength ratio of unity. Apparently the only statements that can be made regarding the significance of the smooth-bar ductility are: (1) Notch sensitivity should be suspected if at a given temperature the smooth-bar ductility decreases rapidly with increasing time to rupture, and (2) Notch sensitivity is often encountered when the smooth-bar ductility is less than 1 or 2 per cent. This latter statement is based on results reported by several German investigators[5] for a series of extremely brittle low-alloy steels which develop a pronounced notch sensitivity when the smooth-bar ductilities drop below 2 or 3 per cent. In contrast, Nimonic 80A at 1200 F, Fig. 15, is not notch sensitive even though the smooth-bar ductility is less than 2 per cent at all rupture times investigated.

INFLUENCE OF THE ALLOY COMPOSITION

Sufficient data are now available from this investigation and also from other published sources to make a tentative appraisal of the relative influence of notches on the stress rupture characteristics of various heat-resistant alloys in their temperature range of application. In the following discussion the effects of composition are summarized as they apply to those conditions of heat treatment usually recommended for the alloy. These heat treatments are presumably those which result in an optimum combination of strength and ductility. It is recognized that the conclusions may have to be modified for other conditions of heat treatment. For the purposes of this discussion the alloys can be divided into three classes: (1) low-alloy ferritic steels, (2) ferritic stainless steels, and (3) austenitic alloys. For the purpose of comparison, the data from the present investigation have been replotted in Fig. 17, which shows the notch and smooth-bar strengths as a function of the test temperature for 1, 10, 100, and 1000 hr to rupture.

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[5] These investigations reported by Thum, Richard, Wellinger, Siebel, and others were recently summarized by Brown and Sachs (3).

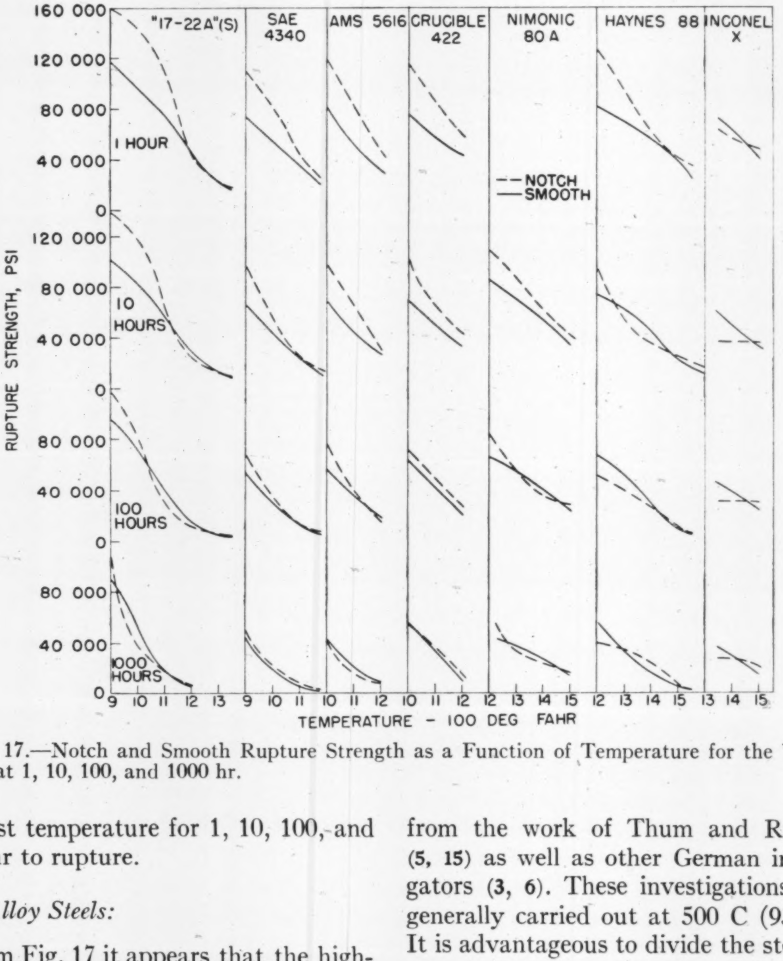

Fig. 17.—Notch and Smooth Rupture Strength as a Function of Temperature for the Various Alloys at 1, 10, 100, and 1000 hr.

Low Alloy Steels:

From Fig. 17 it appears that the high-strength low-alloy steels exhibit notch weakening at temperatures which are considerably lower than ferritic stainless steels or austenitic alloys. The magnitude of the effect varies with the alloy and probably with the heat treatment.[6] Regarding the relative magnitude of embrittlement of alloys in this class, additional information can be obtained from the work of Thum and Richard (**5, 15**) as well as other German investigators (**3, 6**). These investigations were generally carried out at 500 C (932 F). It is advantageous to divide the steels of this class into several types. The basic composition range for these steels in both this country and in Europe appears to be: 0.15 to 0.25 C, 1 to 3 Cr, 0.5 to 1 Mo, and 0.2 to 0.7 Si. Modifications of the basic composition define the following types of steels: (1) Cr, Mo, V steels, (2) Cr, Mo, V, W steels frequently containing larger amounts of silicon and molybdenum, (3) Cr, Mo, Ni steels either with or without vanadium and tungsten, and (4) steels presently under development containing boron and titanium sometimes in combination with vanadium.

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[6] It is now established (**4**) that high normalizing temperatures (above 1800 F) can in many cases reduce the smooth bar elongation of these steels to values less than 1 per cent.

Steels of the basic composition (without additives) are not significantly notch sensitive. Cr, Mo, V steels are notch sensitive, the magnitude of the embrittlement increasing with vanadium content and with decreasing molybdenum and silicon contents. Cr, Mo, V, W steels are generally quite notch sensitive; however, the effect can be reduced by increasing the silicon content up to or slightly greater than 1 per cent. Cr, Mo, Ni steels are particularly notch sensitive, the magnitude of the effect increasing with the nickel content and also with the addition of vanadium and tungsten. Compositions of this type which contain small amounts of nickel can be improved by increasing the silicon and molybdenum contents. There is no published information regarding the effects of notching on the titanium-boron compositions. However, Clark (4) has reported values for smooth-bar elongations which in many cases are less than one per cent.

Ferritic Stainless Steels:

The information available for this class is much more limited than that for the low-alloy steels. For the most part the data are restricted to the 12 Cr types. From Fig. 17 it is apparent that the notch sensitivity in these steels develops at a considerably higher temperature than in the low-alloy ferritic steels. Furthermore, Crucible 422 does not appear to develop significant notch sensitivity under any of the testing conditions. This class of steels generally exhibits relatively high smooth-bar ductilities. However, this *per se* is not an indication of the absence of notch sensitivity (for example, AMS 5616). Thum and Richard (5) report that a 20 Cr, 1 Mo, low-carbon ferritic stainless steel is not weakened by notches at 500 C (932 F) up to 100,000 hr. On the basis of the few tests performed, it appears that the notch embrittlement of these steels is favored by high tungsten and vanadium additions and reduced with increasing Mo content.

Austenitic Alloys:

Very little information is available regarding the notch sensitivity of alloys in this class, including the so-called "super alloys." From Fig. 17 it is evident that while Inconel X and Nimonic 80A are quite notch sensitive at 1300 and 1400 F "recovery" has occurred at the preferred temperature of application (1500 F). At this temperature the alloys would presumably not be weakened by notching regardless of the time to failure. The boron-modified hot cold-worked stainless steel, Haynes 88, is very notch sensitive at temperatures between 1200 and 1500 F. It should be noted here that according to Siegfried (7), cold deformation definitely increases the notch sensitivity of the super alloy G18-B at 1200 F. Siegfried (8) has reported additional results on this alloy which indicate it to be notch sensitive at 650 C (1200 F) and 700 C (1290 F) in the solution-quenched condition.

SIGNIFICANCE OF RESULTS

The rather extensive tests reported here indicate that a variety of high-strength heat-resistant alloys may be weakened in creep loading by a sharp notch. These findings are supported by several other investigators both in this country and in Europe.

It has now been established that the notch effect observed in stress rupture tests is time-temperature dependent both in magnitude and in its temperature range of occurrence. In addition, the results indicate that the smooth-bar ductilities can yield no definite information regarding the notch sensitivity of a given alloy.

The question now arises as to how the results of such tests are to be interpreted by the designer. In their present form the data definitely indicate that the effects of stress concentration cannot be ignored in the design of parts from many high-strength alloys. On the other hand, contrary to the authors' previous conclusions (1), certain alloys (SAE 4340 and Crucible 422) are for practical purposes free from notch sensitivity even for the extremely high elastic stress concentrations used in this investigation. The consideration of the influence of stress concentrations becomes particularly important in certain severe service applications where weight or other limitations require that extremely small margins of safety be used. For example, in the case of Inconel X, Fig. 18, if a rather generous factor of safety of 1.5 were used, the design stress for 100-hr life at 1350 F would about equal the sharp notch rupture strength of the material.

Important additional information which is now extremely limited concerns the influence of the magnitude of the stress concentration. This can probably best be obtained by investigating the effects of notch sharpness as was done recently by Davis and Manjoine (9) for a few alloys.[7] If the elastic stress concentration factor and average stress for a part configuration is known, then it should be possible to arrive at a good approximation of the life at a given temperature from experimentally determined curves relating the notch strength to the notch sharpness. A comparison between the effects of notch sharpness observed in stress rupture tests and in room temperature tension tests (3, 6) has shown the effects of sharpness in these two tests to be essentially similar for the alloys thus far investigated.

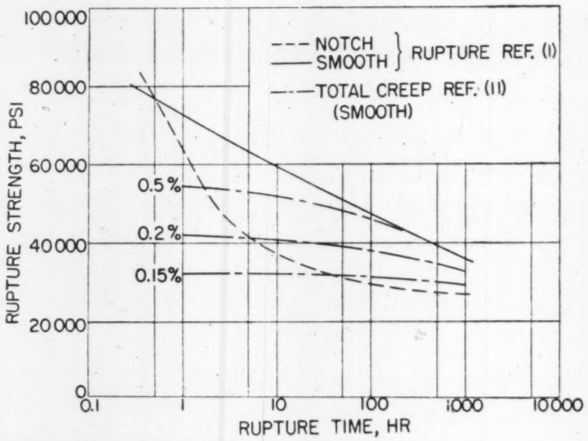

FIG. 18.—Comparison of Notch and Smooth Rupture Strength with Total Creep for Inconel X at 1350 F.

A further consideration is based on those service applications where the limiting factor is the time to reach a certain amount of total creep at a given stress and temperature. Total creep data are rather difficult to find in the literature for the alloys reported here. However, such data for "17-22A" (S) disks have been reported by Freeman (**10**) and the manufacturers' data books give data for Inconel X (**11**) and Nimonic 80A (**12**). These data have been plotted in Figs. 8, 18, 19.[8] For these alloys, a design for certain total deformation would be limited by the effects of a stress concentration. For example, the curves for 1 per cent total creep "17-22A" (S) at 1000 and 1100 F, Fig. 19, intersect the notch rupture-strength curves at times of approximately 200 and 30 hr, respectively, and at higher times the notch rupture strength is below the stress for 1 per cent total deformation. Thus, it would be possible to fracture a part in a section containing a stress concentration before the limiting deformation was reached in an adjacent section (assuming the average stress in both sections to be the same). This situation is aggravated if a notch sensitive alloy possesses extremely high resistance to creep. This is the case for both Inconel X and Nimonic 80A, Figs. 18 and 8. The creep data shown for these alloys at temperatures of 1350 and 1400 F, respectively, indicate that the effects of stress concentrations should be considered when limiting total deformations are greater than a few tenths of a per cent.

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[7] It should be mentioned that Davis and Manjoine have shown notch weakening in the alloy K-42-B for elastic stress concentration factors only slightly greater than two. This would correspond to a 0.024-in. radius notch in the specimens used in this investigation.

While no definite evidence is yet available, it is felt that notch rupture tests should reveal qualitative information regarding the relative susceptibility of an alloy to embrittling factors other than notches. This speculation is supported by a considerable amount of evidence accumulated in the study of short time tensile and impact properties of low alloy steels. Thus, it has been pointed out by Sachs (**13**), Ripling (**14**), and others that steels which are notch sensitive in room-temperature static tests are also those subject to embrittlement in impact or tensile loading (without notches) at low temperatures. In creep loading, the notch specimen appears in every case to fail in an intercrystalline manner. There-

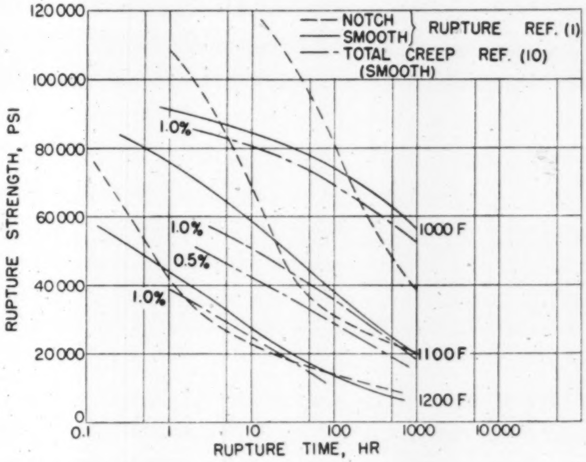

Fig. 19.—Comparison of Notch and Smooth Rupture Strength with Total Creep for "17-22A" (S) at Several Temperatures.

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[8] In all cases the reported rupture data agreed well with that shown in this paper, and it would seem reasonable to assume the creep data would also be representative of the alloys tested in this investigation.

fore the notch strength should be an indication of the strength of the crystal boundaries under conditions of high stress and restraint. These conditions can occur in other types of high-temperature loading during service.

Acknowledgment:

The authors wish to acknowledge the helpful discussions with George Sachs, Consultant to National Advisory Committee for Aeronautics, S. S. Manson, and B. Pinkel of the Lewis Laboratory of the NACA. Also appreciation is expressed to O. T. Hickey and S. P. Barnosky for their able assistance with the tests.

Thanks are due the various manufacturers who cooperated to the fullest extent in making available the special alloys tested. Also appreciation is expressed to J. W. Freeman of the University of Michigan and the Wright Aircraft Development Center for permission to report the creep data for the "17-22A" (S) disks.

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TIME-TEMPERATURE DEPENDENCE OF THE NOTCH EFFECT AND INFLUENCE OF NOTCH DEPTH IN STRESS RUPTURE TESTS ON A Cr-Mo-V STEEL*

By D. P. Newman,[1] M. H. Jones,[1] and W. F. Brown, Jr.[1]

Synopsis

Notch and smooth bar stress-rupture tests were carried out on the Cr-Mo-V steel "17-22A" (S) over a wide range of temperatures from 600 to 1350 F. This systematic series of tests was designed to yield precise information on the time-temperature dependence of the notch effect in this low-alloy steel. The specimens possessed 50 per cent, 60 deg, sharp V notches and were tested for times up to 1000 hr.

It was found that the trends of both the notch rupture - strength ratio and the notch ductility, when plotted against testing temperature with time as a parameter, conform with the previously advanced hypothesis that a precipitation reaction is associated with the notch sensitivity. A further analysis of these data yields an activation energy for the process which is close to that obtained for diffusion of C in alpha iron. Hardness values taken from the heads of the broken bars indicate that precipitation can be induced by heating alone.

The same steel was used to investigate the influence of notch depth at 1100 F. Several depths between 5 and 84 per cent were selected, and sufficient tests were made to determine the influence of depth at times between approximately 0.1 and 300 hr. The results indicate a strong similarity between the behavior of a notch ductile alloy in stress-rupture tests and in room temperature tension tests. For the notch brittle conditions, it was found that the depth at which the onset of notch sensitivity occurred is shifted to lower values with an increase in the testing time. In addition, for rupture times exceeding a certain value, it appears that any sharp notch, however shallow, results in weakening.

Based on tests performed over a limited range of stress and temperature but on a wide variety of alloys, the authors (**1**)[2] have suggested that the weakening effect of notches observed in stress rupture tests is a time-temperature dependent phenomenon. In addition, it has been postulated that the notch sensitivity observed in a number of heat-resistant alloys may be related to structural changes taking place during the course of the test.

It is rather difficult to examine these postulates critically in light of the evidence presented to date in the literature. In general, no systematic series of notch rupture tests that covers a wide range of both stress (time) and temperature has been carried out on a single alloy.

Furthermore, there appears to be no

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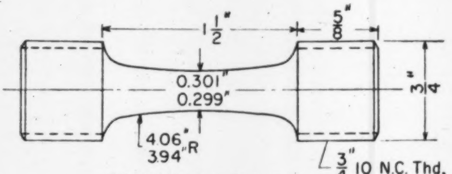
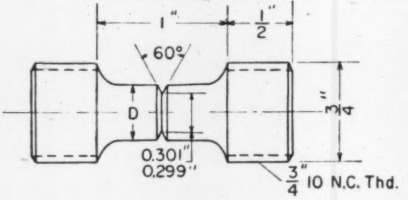
[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 689.

direct evidence to indicate a correlation between microstructural changes occurring in a creep test and notch rupture sensitivity. However, several investigators including Smith *et al.* (2) and Miller *et al.* (3) report that low-alloy steels subject to long-time exposure at elevated temperatures undergo a precipitation which is presumably associated with the presence of molybdenum. In addition, it has been shown (2) that the high-temperature exposure of these steels may reduce the transition temperature in impact tests. Several investigators including Siegfried (4), Davis and Manjoine (5), and the authors (1) have made studies of the microstructure of fractured notched specimens using conventional techniques. The results of such investigations cannot be directly correlated with the observed embrittlement in the notch test. These studies are extremely difficult since it is probable that the quantity of material involved is very small and that the maximum effects on the mechanical properties would occur when this material was in a very fine state of dispersion. In addition, the matrix of many of the alloys, particularly the low-alloy steels, is filled with carbides, formed during the heat treatment which may mask other precipitated phases.



(a) Unnotched Specimen



Notch Radius Less Than 0.001 in.

Notch Depth	D
5%	0.308"
20%	0.335"
35%	0.373"
50%	0.424"
60%	0.474"
70%	0.548"
84%	0.750"

(b) Notched Specimen

FIG. 1.—Stress-Rupture Specimens.

An understanding of the complex phenomena involved in high-temperature embrittlement and notch sensitivity requires, in addition to the above mentioned studies, a knowledge of the effects of the geometrical variables of the notch. The effects of notch sharpness in stress rupture tests have been investigated by Siegfried (6) and by Davis and Manjoine (5). It has been pointed out (7)[3] that, on the basis of the tests made thus far, the influence of this geometrical variable on the fracture characteristics in creep loading is essentially similar to that observed in room temperature tension tests. On the other hand, a similar analysis of the effects of notch depth cannot be made on the basis of the very limited tests reported in the literature. Siegfried (6) has reported data for three notch depths (at four sharpnesses) for a tin-cadmium alloy tested at room temperature. A few tests at 1200 F on 19-9DL alloy for one stress level and for three notch depths were reported by Davis and Manjoine (5). In contrast, extensive room temperature investigations have been made by Sachs, Lubahn, and Ebert (8) which clearly indicate the effects of notch depth and notch sharpness in tension tests on low-alloy steels.

The present investigation was designed to reveal fundamental information regarding the following: (1) a more precise knowledge of the time-temperature dependence of the notch effect in stress rupture tests, (2) evidence for structural changes accompanying the creep process which might account for the embrittlement, and (3) the influence of the notch depth on the notch strength over a wide range of stresses at one test temperature. It was felt that a systematic series of tests on one alloy covering a wide range of the experimental variables would contribute best to an understanding of the basic phenomena involved. The alloy selected was a high strength chromium-molybdenum-vanadium steel developed by Timken and designated as "17-22A" (S). This material is highly uniform and is representative of a large class of creep-resistant low-alloy steels now of great interest and undergoing rapid development.

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[3] W. F. Brown, Jr., D. P. Newman, and G. Sachs, discussion of paper by Davis and Manjoine (5).

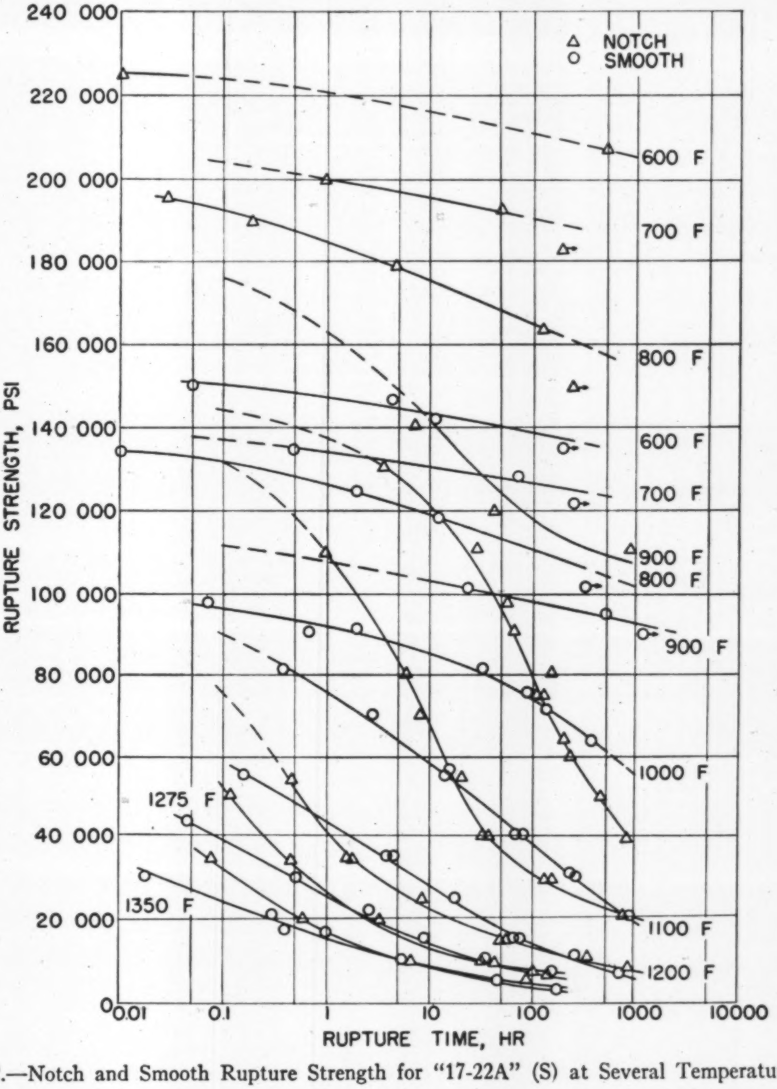

FIG. 2.—Notch and Smooth Rupture Strength for "17-22A" (S) at Several Temperatures.

MATERIAL AND PROCEDURE

The "17-22A" (S) steel used in this investigation had the following composition: 0.30 C, 0.58 Mn, 0.015 P, 0.020 S, 0.63 Si, 1.24 Cr, 0.21 Ni, 0.50 Mo, 0.12 Cu, and 0.25 V. Specimen blanks were cut from 1 in. diameter hot-rolled bar stock. These blanks were normalized at 1725 F $\frac{1}{2}$ hr and air cooled, and then tempered at 1200 F for 6 hr and air cooled. The Rockwell C hardness was 33. Specimen blanks for the investigation of notch depth were heat treated in one group.

Specimen types are shown in Fig. 1. Investigations of time-temperature dependence of the notch strength were made using a notch depth of 50 per cent. Various notch depths were obtained by holding the notch diameter to 0.300 in. and varying the diameter of the cylindrical section to produce depths between 5 and 84 per cent. For all tests the notch radius was machined with a lathe tool to 0.001 in. or less.

The testing procedure and equipment have been previously described (1). In the investigation of notch depth, universal joints were inserted between the specimen loading rods and the heads of the creep machines. This procedure resulted in a considerable reduction in the eccentricity of loading (9) and consequently reduced the scatter in the notch strength values.

For the investigation of notch depth, seven stresses were selected. The range of stress was sufficiently wide to be representative of the behavior of the alloy at 1100 F in both the notch-brittle and the notch-ductile conditions. The previously mentioned constant notch diameter (equal to that of the minimum diameter of the smooth bars) was maintained to minimize the effect of section size.

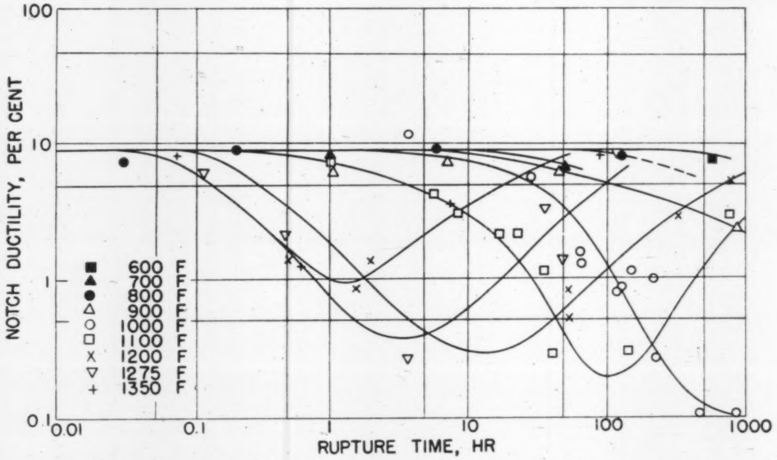

FIG. 3.—Notch Ductility for "17-22A" (S) at Several Temperatures.

The Rockwell A hardness was measured on 0.25-in. diameter cylindrical specimens machined from the heads of the fractured rupture specimens used in the investigation of time-temperature dependence of the notch effect. The hardness values reported represent an average of five readings.

TIME-TEMPERATURE DEPENDENCE OF THE NOTCH EFFECT

The results of smooth and notch bar tests on "17-22A" (S) at 600, 700, 800, 900, 1000, 1100, 1200, 1275, and 1350 F[4] are assembled in Figs. 2 and 3. Notch and smooth bar strengths are plotted in Fig. 2; notch bar ductilities are shown in Fig. 3.

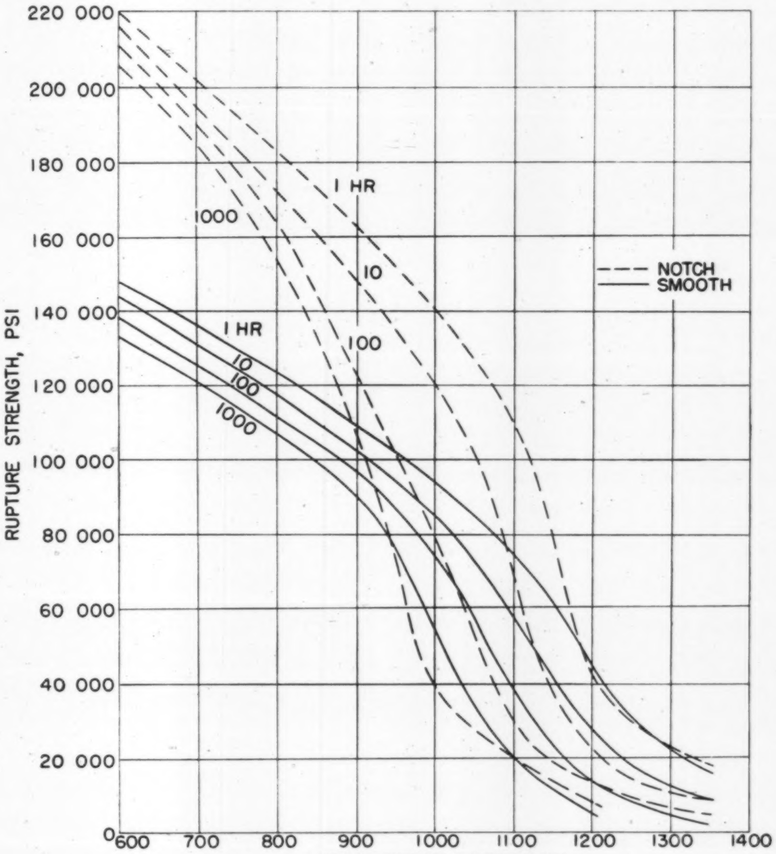

FIG. 4.—Notch and Smooth Rupture Strength of "17-22A" (S) as a Function of Temperature for Various Rupture Times.

From Fig. 2 it is established that both the magnitude of the notch sensitivity and its time range are temperature dependent. The notch sensitivity decreases with increasing temperature, and at the highest test temperature the notch strength curve is tangent to the curve for the smooth bars. At still higher temperatures, presumably only notch strengthening would be observed. The time of onset of notch sensitivity is shorter, the higher the temperature, up to a temperature between 1275 and 1350 F. At still higher temperatures, the onset of notch sensitivity shifts to longer times. At sufficiently long times to rupture (at any temperature), the notch weakening disappears and notch strengthening occurs (for example, at 1200 and 1275 F). This "recovery" occurs earlier with increasing temperature.

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[4] The data at 600, 700, and 800 F were obtained by Sachs (unpublished research).

Some further insight into the behavior described above can be obtained if the results are replotted with test temperature as the independent variable and rupture time as parameter (Fig. 4).

Except at the highest testing temperatures, the rate of decrease of strength with temperature is greatest for the notched bars. This difference in rate is most pronounced in the temperature range between 1000 and 1200 F. In contrast, at the highest testing temperatures this trend is reversed, and the notch strength changes less with temperature than the smooth bar strength. This latter behavior in combination with the decrease in the magnitude of the notch sensitivity with increasing temperature explains the observed shift in the onset of notch sensitivity to longer times above a certain temperature (Fig. 2). In addition, the decrease in the magnitude of the notch sensitivity with increasing temperature and decreasing time indicates that for very short times

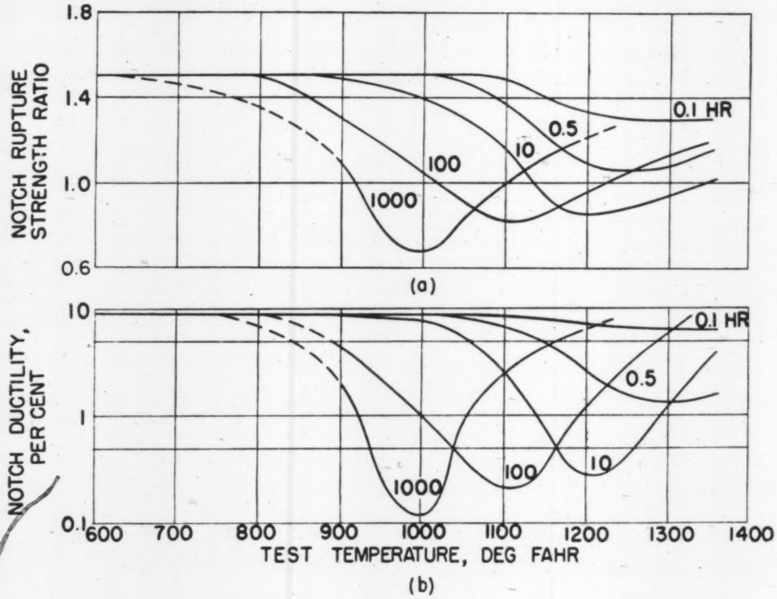

FIG. 5.—Notch Rupture Strength Ratio and Notch Ductility for "17-22A" (S) as a Function of Test Temperature for the Rupture Times Indicated.

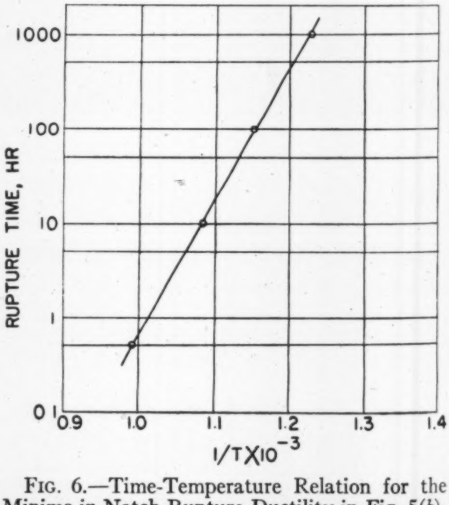

FIG. 6.—Time-Temperature Relation for the Minima in Notch Rupture Ductility in Fig. 5(*b*).

to rupture no notch sensitivity would be observed at any temperature.

The time-temperature dependence of the embrittlement is also reflected in a plot of the notch bar ductilities against the time to rupture with temperature as parameter (Fig. 3). These curves bear a strong similarity to those obtained when the room temperature tensile ductility of an age-hardening alloy is plotted as a function of the aging time with aging temperature as a parameter.

The effects of time and temperature on strength discussed above can be best represented by plotting the ratio between the strengths of the notch and smooth bars (notch rupture - strength ratio) against the testing temperature, with rupture time as parameter (Fig. 5(*a*)).

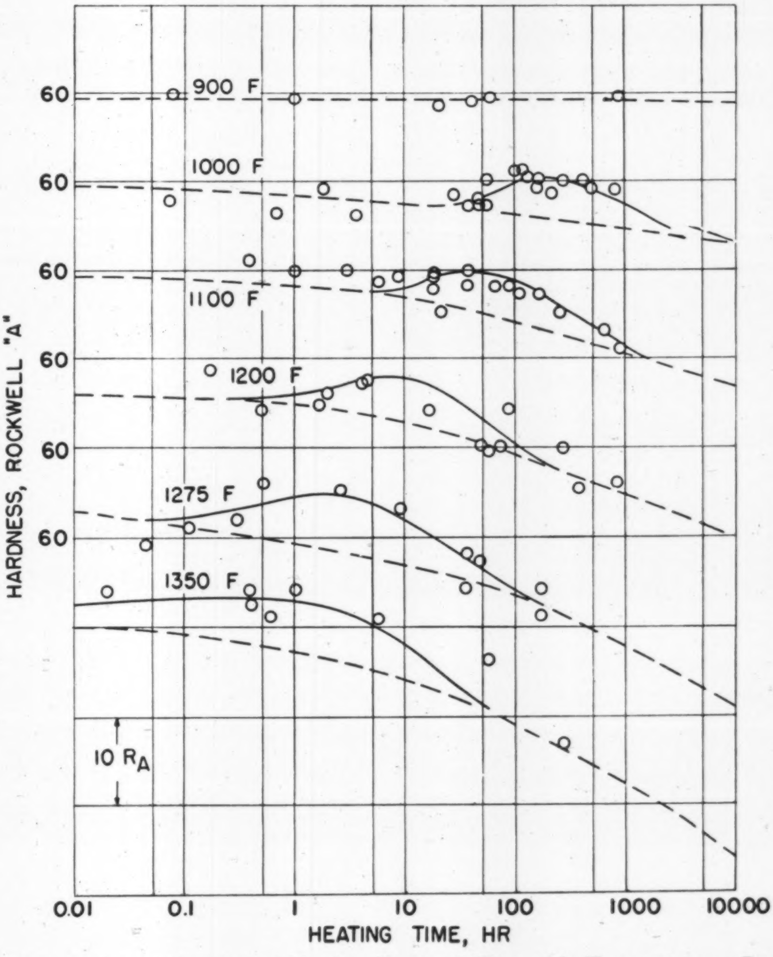

FIG. 7.—Hardness as a Function of Heating (Rupture) Time with Temperature as Parameter for "17-22A" (S).

This representation differs from that previously formulated (1) on the basis of more limited data. It now appears that a constant notch rupture - strength ratio of 1.5 is developed for sufficiently short times to rupture at any temperature. This value of maximum strengthening is in agreement with that predicted theoretically for this notch geometry, and with that obtained in room temperature tension tests on low-alloy steels (**10, 11**). For a given rupture time, the notch rupture-strength ratio deviates downward from a value of 1.5 at a temperature which is higher the shorter the rupture time. At a given time the notch rupture-strength ratio passes through a minimum which occurs at progressively lower temperatures and is deeper with increasing rupture time. The recovery of the notch rupture-strength ratio from the minimum value is most pronounced at the longest times to rupture. Figure 5(*b*) shows a plot of the notch ductility as a function of the test temperature. It is noted that these curves are very similar to those obtained in Fig. 5(*a*) for the notch rupture-strength ratio.[5]

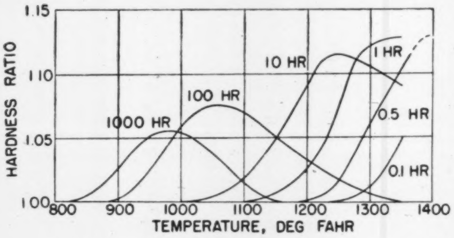

FIG. 8.—Hardness Ratio as a Function of Test Temperature at Several Heating Times (Times to Rupture) for "17-22A" (S).

These representations give support to the previously advanced hypothesis that notch rupture sensitivity in this alloy is associated with a structural change such as a precipitation. An estimate may be made of the activation energy for the structural change from the trend of the notch rupture ductility curves in Fig. 5(*b*). If it is assumed that the minima in the notch ductility represent equivalent stages in the precipitation process, then the temperatures and times at the various minima should be related by the rate equation:

$$t\,ke^{-Q/RT} = C$$

where:

t = rupture time,
T = absolute temperature,
R = gas constant,
Q = activation energy, and
k and C = constants.

The equation implies that a plot of log t *versus* $1/T$ should be linear and that the slope of the line is equal to the activation energy. This plot (Fig. 6) yields an activation energy of approximately 25,000 cal per mol. This compares with 20,000 cal per mol obtained by Wert (**12**) for the diffusion of carbon in α iron. The value of 25,000 cal per mol reported here is only approximate, since the positions of the minima in the ductility curves are not precisely established. In comparing values it should be remembered that the energy determined by Wert was obtained in the absence of plastic strain and at lower temperatures. While the temperatures and times at the ductility minima are related by the rate equation, the magnitude of the embrittlement and therefore the notch strength would not be expected to follow a simple time-temperature relation. Thus the precipitate exerts a variable effect on the strength depending on its structure, which is in turn determined by both time and temperature.

In spite of the uncertainties still involved, it is believed that the observed effects are best explained by a diffusion of carbide in the α iron matrix. This steel contains strong carbide formers, and it has been noted (**9**) that the degree of embrittlement increases with the addition of carbide forming elements.

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[5] The influence of ductility on the retained stress concentration as well as the influence of the stress state in notch tests at elevated temperatures has been discussed previously in detail (**1, 7**).

The precipitation of carbides could act in two ways to reduce the ductility: (1) it could strengthen the grains and thereby inhibit grain boundary movement which would normally act to relieve the stress concentration, and (2) it could embrittle the grain boundaries and thereby cause them to fracture at a point of high stress concentration. The emphasis has been placed on the rôle of the grain boundaries, since metallographic examination indicates that notch rupture sensitivity is associated with intercrystalline fractures. However, the mere presence of intercrystalline fractures does not indicate notch sensitivity, since such fractures also occur in regions of notch strengthening observed when the notch strength recovers at long times to rupture. In order to gain a further understanding of the complex embrittling mechanism, tests are needed

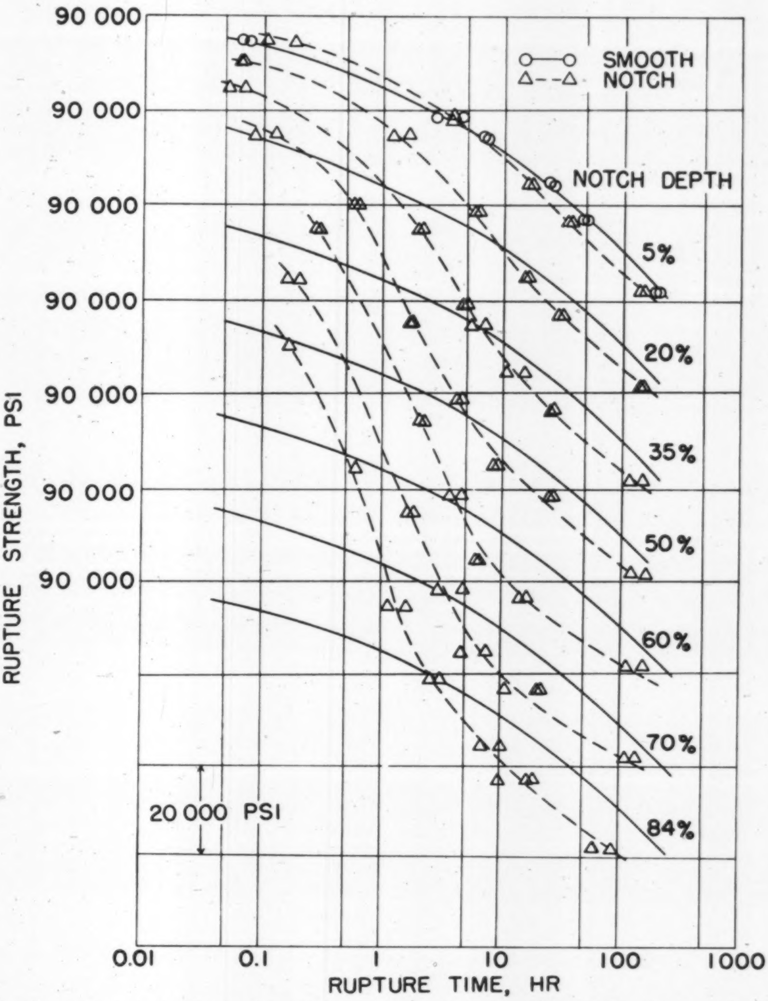

FIG. 9.—Smooth and Notch Bar Rupture Strengths at Several Notch Depths for "17-22A" (S) Tested at 1100 F.

that will directly indicate the rôle of stress in the embrittlement occurring with progressive time at a given temperature, and that will yield information regarding the development of failure in notch bars.

HARDNESS STUDIES

The hardness of the heads of the specimens is shown in Fig. 7 as a function of rupture time with temperature as a parameter. Since the heads are strain free, the hardness changes are due to heating alone. The curves show considerable scatter, particularly at the higher testing temperatures, for two reasons: (1) The heads of the specimens varied in temperature from the test section by an amount which depended on the type of furnace. This variation in some cases was 25 F. (2) The heating time before loading was between 2 and 3 hr. This variation would be expected to be significant only for the short rupture times at the higher temperatures.

In spite of the scatter and the relatively small changes in hardness, definite trends in the curves are evident. If only agglomeration of the structure occurred during heating, then the hardness at a given temperature should decrease continuously with increasing time and the rate of decrease at a given time should increase with temperature. The dotted curves in Fig. 7 have been drawn in conformance with this basic behavior. These curves were derived from a plot of the hardness as function of temperature with time as parameter. In spite of the scattering, it is felt that the hardness data show deviations from these basic curves which might be expected if precipitation hardening occurred. In order to compare these hardness variations with time-temperature variations of notch sensitivity, the ratio between the measured hardness and that taken from the dotted curves at a corresponding time has been plotted against temperature for times of 0.01, 0.5, 1, 10, 100, and 1000 hr. These curves, Fig. 8, exhibit maxima that shift to lower temperatures with increases in time. Also, the magnitude of the maxima is greatest at the highest temperatures (shortest times). Comparing Fig. 5(*a*) with Fig. 8, it is seen that the temperature of maximum hardening is nearly coincident with that of the maximum notch sensitivity for times of 100 and 1000 hr. At shorter times the minimum in the notch strength ratio occurs at temperatures lower than that of maximum hardening. In addition, it appears that the maximum notch sensitivity is produced at times that yield the smallest changes in hardness.

EFFECT OF NOTCH DEPTH

The data obtained at 1100 F using various notch depths are assembled in Fig. 9, which shows the notch and smooth bar strengths as a function of rupture time with notch depth as parameter.

These curves are similar in that a transition from notch strengthening to notch weakening occurs for all notch depths investigated as the time increases. However, the magnitude of both the strengthening and the weakening vary with the depth. Moreover, the time of onset of notch weakening appears to decrease with increasing notch depth.

In order better to visualize the effects of notch depth in stress rupture tests and to permit comparison with those observed in room temperature tension tests, the notch rupture-strength ratios have been plotted in Fig. 10 as a function of the notch depth with rupture time as a parameter.

At short rupture times the notch rupture-strength ratio first increases with notch depth and then gradually decreases. The rate of increase is less and the maximum values attained are lower with increasing time to rupture. If the rupture time is sufficiently long (5 hr), the notch-strength ratio decreases to values less than unity, after passing through a maximum. At longer rupture times, the notch-strength ratio continuously decreases with increasing depth to a nearly constant value at depths greater than approximately 60 per cent.

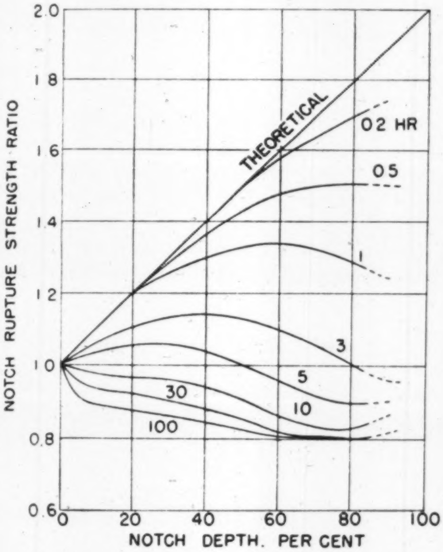

FIG. 10.—Notch Rupture Strength Ratio as a Function of Notch Depth with Rupture Time as Parameter for "17-22A" (S) at 1100 F.

In general, the curves decrease in level with increasing time to rupture. However, at notch depths greater than 60 per cent, the notch-strength ratios at 30 and 100 hr to rupture are identical. This might be expected, since with increasing rupture time the notch strength would eventually recover (Fig. 2). Thus a rise in the level of the curves shown in Fig. 10 would occur for times exceeding a certain value. The nature of the notch rupture-strength ratio curves in this time range cannot be determined on the basis of the present tests since the effect of notch depth on the recovery time is not known.

The influence of notch depth in stress rupture tests may be compared with the influence of this variable in room temperature notch tension tests. Sachs, Lubahn, and Ebert (8) determined the notch tensile strength as a function of the notch depth for SAE 3140 heat treated to various strength levels. For purposes of comparison, notch tension data have been selected at three strength levels, 145,000, 220,000, and 240,000 psi, corresponding to tempering temperatures of 1000, 740, and 650 F, respectively. In Fig. 11(*a*), two notch ductile conditions of this steel (145,000- and 220,000-psi strength levels) are compared with two notch ductile conditions of "17-22A" (S) in rupture tests (0.2 and 1.0 hr). A similar comparison for notch brittle conditions is made in Fig. 11(*b*) using the 240,000-psi strength level steel and the 100-hr rupture tests.

The curves for rupture tests and those for tension tests are basically similar in several respects: (1) The notch strength ratios for the most ductile conditions in either test exhibit a continuous increase with increasing depth. This increase follows the theoretical linear relation (8) up to high notch depths. (2) The deviation from theoretical behavior occurs at lower depths for conditions possessing lower notch ductilities. (3) The notch sensitive conditions possess very low notch ductilities.

On the other hand, there are two definite dissimilarities: (1) The effect of the decrease in the effective stress concentrations at high notch depths does not produce a pronounced increase in the notch-strength ratio of the rupture tests, as is observed for the 240,000-psi steel. (2) The 100-hr rupture tests exhibit a decrease in notch-strength ratio with increasing depth for any depth however shallow. This is in contrast to the 240,000-psi steel which is notch strengthened at depths less than 30 per cent.

Summary of Results

On the basis of the tests reported here, the following statements may be made regarding the influence of time, temperature, and notch depth on the notch stress rupture characteristics of the low-alloy Cr-Mo-V steel "17-22A" (S):

1. Tests covering a wide range of temperature and stress indicate that notch sensitivity in this alloy is a complex function of both time and temperature. In the temperature range of application (1200 F), a 50 per cent, sharp, 60 deg "V" notch produces approximately 50 per cent increase in the strength of this alloy at sufficiently short times to rupture. With increasing rupture time at a given temperature, the notch strengthening is gradually reduced and replaced by notch weakening. The weakening effect develops to a maximum and then decreases. At sufficiently high rupture times, notch strengthening is again observed. With increasing test temperature, the time of onset of notch weakening is reduced and the magnitude of the effect decreases.

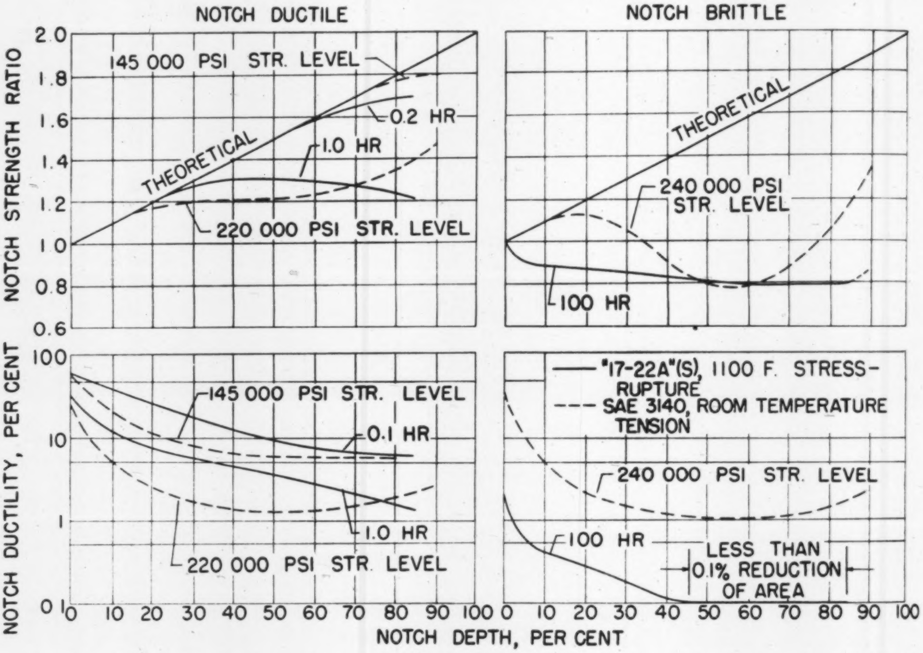

Fig. 11.—Notch Strength Ratio and Notch Ductility as a Function of Notch Depth for 1100 F Stress-Rupture of "17-22A" (S) and for Room Temperature Tension Tests of SAE 3140.

2. An analysis of both the notch strength and notch ductility data strongly suggests a precipitation of carbide is associated with notch sensitivity in this steel and that the precipitate acts to cause retention of a portion of the high initial elastic stress concentration at failure.

3. A study of the changes in hardness with heating time and temperature reveals that this steel undergoes a slight hardening effect with increasing time at a given temperature. This effect develops to a maximum at a time that is shorter the higher the temperature.

These variations in hardness lend further support to the concept that a precipitation reaction is associated with notch sensitivity in this alloy.

4. For notch ductile conditions of the alloy (rupture times less than 1 hr at 1100 F), the effect of increasing notch depth is to increase continuously the notch-strength ratio.

5. At 1100 F the time of onset of notch weakening occurs at slightly reduced times with increases in notch depth.

6. At rupture times yielding the maximum notch sensitivity at 1100 F, any notch however shallow results in notch weakening.

Acknowledgments:

The authors wish to thank George Sachs, consultant to the National Advisory Committee for Aeronautics, for his helpful criticism and advice during the course of the program, and also for furnishing additional data on "17-22A" (S). Appreciation is expressed to B. Pinkel and S. S. Manson at the Lewis Laboratory of NACA for their interest and encouragement. The authors are also indebted to S. P. Barnosky and O. T. Hickey for their assistance with the tests. C. L. Clark of the Timken Roller Bearing Co. was extremely helpful in obtaining the material used in the investigation.

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DISCUSSION

Mr. W. Siegfried.[1]—The authors of this paper have succeeded in establishing systematic relationship between various factors connected with the embrittlement of metals in long-time creep tests. Two points in particular are put forward in a very convincing way, namely, that embrittlement at high temperatures is dependent on time and that certain definite relations exist between precipitation reactions and embrittlement.

The results published here are confirmed by the experience carried out in the laboratory of Sulzer Brothers at Winterthur.[2] There also it was observed that in long-time creep tests on notched specimens and on models of blade-root connections of austenitic steels the notch effect first rose with increasing times and then dropped off at even longer times, this being the case both with the test specimens and with the models. The same phenomenon was observed in long-time creep tests carried out with tin-cadmium alloys at room temperature. It thus seems that there is some general law behind the phenomena observed by the authors in a chromium-molybdenum-vanadium steel. In our own tests on an austenitic chromium-nickel-cobalt steel of the following analysis:

Carbon, per cent	0.4
Manganese, per cent	0.8
Silicon, per cent	1.0
Nickel, per cent	13
Chromium, per cent	13
Tungsten, per cent	2.5
Molybdenum, per cent	2
Niobium, per cent	3
Cobalt, per cent	10

we also found that the notch sensitivity in the creep test is associated with precipitation reactions. We also established, it is true, that the rate of the precipitation processes, which was determined from the change in the properties at room temperatures, depended greatly on the stress. In the tests with the tin-cadmium alloys at room temperature it was not possible to obtain any further evidence on precipitation processes, so that the question is still open as to whether embrittlement at high temperatures is also due to other processes.

Metallographic investigations, though carried out so far only on austenitic steels, have shown that notch embrittlement at high temperatures is due to the deep penetration of an intercrystalline crack, while in non-brittle steels cracks which had formed were later rounded out by the creep process.

It is interesting in this connection to draw attention to tests on smooth bars carried out by Tapsell.[3] These were long-time creep tests covering a wide temperature range on a molybdenum-vanadium steel with 0.14 per cent carbon, 0.54 per cent molybdenum and 0.2 per cent vanadium. The accompanying Fig. 12 shows the results of Tapsell's investigations.

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[1] Sulzer Brothers, Ltd., Winterthur, Switzerland.

[2] W. Siegfried, " Investigations into the Influence of Notches on Creep Strength at High Temperatures," Symposium on Strength and Ductility of Metals at Elevated Temperatures, with Particular Reference to Effects of Notches and Metallurgical Changes, p. 93, Am. Soc. Testing Mats. (1953). (Issued as separate publication *ASTM STP No. 128.*)

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[3] H. J. Tapsell, "Behaviour of Metals at High Temperatures with Special Reference to Creep," *De Ingenieur No. 41*, Materialenkennis.

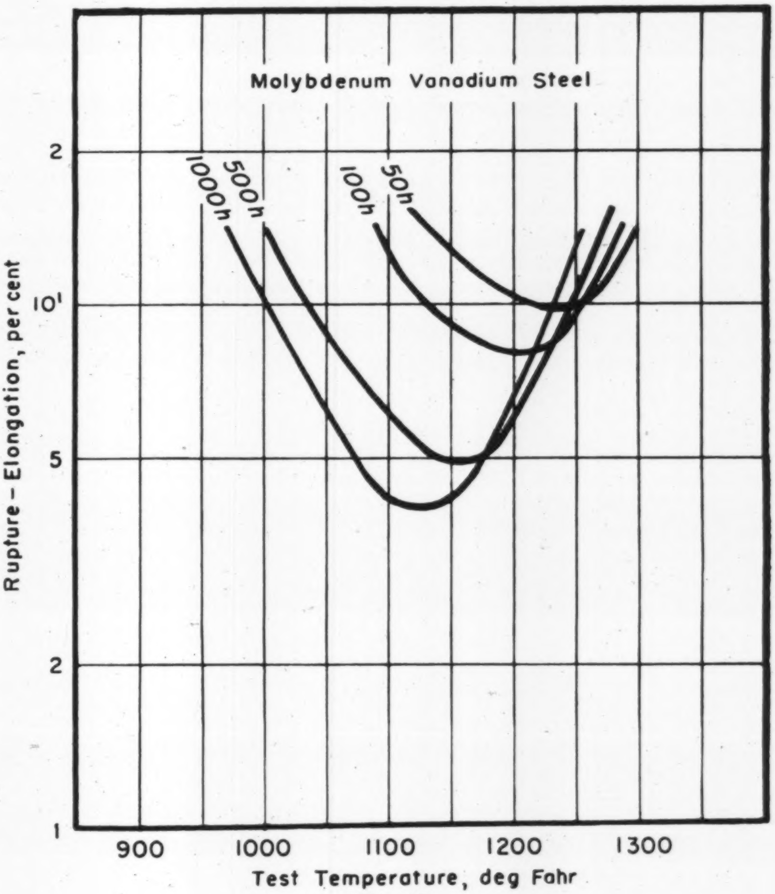

FIG. 12

MR. D. P. NEWMAN, M. H. JONES AND W. F. BROWN, JR. (*authors' closure*).—The authors wish to thank Mr. Siegfried for his interesting discussion and appreciate his comments that tests performed at the Sulzer Bros. laboratory confirm the general conclusions set forth in this paper.

Regarding Mr. Siegfried's comment that his tests on tin-cadmium alloys did not yield evidences of precipitation phenomena, we feel that this material was not subject to the very large embrittlement often observed in the steels. However, we do not disagree with Mr. Siegfried's statement that creep embrittlement may involve other processes than precipitation. We simply wish to emphasize that the results observed strongly indicate that a precipitation may contribute toward the embrittlement.

The authors agree with Mr. Siegfried that a material with strong grains and relative weak grain boundaries will possess a tendency toward embrittlement in the creep test and to notch rupture sensitivity. This is essentially one of the postulates stated in the present paper.

The results of Tapsell on a chromium-molybdenum-vanadium steel are quite interesting and show a time-temperature dependence for the elongation which is very similar to that shown by the authors for reduction of area values.

TIME-TEMPERATURE-STRESS RELATIONS FOR THE CORRELATION AND EXTRAPOLATION OF STRESS-RUPTURE DATA*

By S. S. Manson[1] and W. F. Brown, Jr.[1]

The problem of correlating and extrapolating stress rupture data has recently received considerable attention. In general, there have been two approaches. The methods proposed are based either on certain known fundamental relations or the data are treated empirically.

Machlin and Nowick (**1**)[2] derived a relationship among stress, temperature, and time-to-rupture based on the rate process theory. This simple explicit relation among the three variables predicted linearity of stress *versus* log of rupture time at a constant temperature. Deviations from linearity were interpreted as indications of structural instabilities. Because nearly all heat-resisting alloys exhibit precipitations or other forms of structural instability in their temperature range of service, the Machlin and Nowick relation has received only limited application. Servi and Grant, (**2**) have made an analysis of creep data on the alloy S-590 in which it is concluded that the rate process theory is not suitable for the correlation of creep data when the range of creep rates is large.

The method of Larson and Miller (**3**) is based on the assumed linearity of plots of log rupture time against reciprocal of absolute temperature at a constant nominal stress, and the convergence of such lines to a point, $-C$, on the time axis (that is, at infinite temperature). Thus at a constant nominal stress $(T + 460)(\log t + C)$ should be a constant, where:

T = temperature in degrees Fahrenheit and

t = rupture time in hours.

Based on the examination of a large number of materials, they concluded that a suitable universal value of C is 20. Thus a plot $(T + 460)(20 + \log t)$ *versus* stress should give a single "master" curve, irrespective of the individual values of stress, time, and temperature. Available data in the low- and medium-time range should then be usable for the prediction of the longer times. Manson and Haferd (**4**) have made a critical analysis of the Larson and Miller parameter and have indicated that considerable error may result when this parameter is used for extrapolation purposes. These errors arise because of deviations from linearity of plots of log t *versus* $1/(T + 460)$ as well as the lack of tendency for such plots to converge to a single point on the log t axis.

In the method of Hoff (**5**) account is taken of the continuous increase in stress on a specimen under constant load resulting from changes in cross-sectional area accompanying creep. The hypothesis is that the change of curvature of a conventional creep curve from linear to concave upward is due to this increase in stress and that the rupture time can be approximated by computing the theoretical time at which the cross-sectional area approaches zero. The relation between steady (minimum) creep rate and true stress must be known in order to make the computation. By neglecting transient creep and making use of the approximate linearity plots of stress *versus* steady creep rate on log-log coordinates, the predicted rupture times for an aluminum alloy were found to agree very well with the experimental rupture times. The practical utilization of the method does, however, depend on the availability of extensive creep tests, which generally are more difficult to perform than stress-rupture tests, and on the validity of the dimensional instability criterion of rupture. Since in some cases rupture occurs with relatively little gross contraction in cross-sectional area and since at elevated temperatures intercrystalline cracking may account for the initiation of third stage creep, it is expected that the method may have limitations.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Nat. Advisory Com. Aeronautics, Lewis Flight Propulsion Laboratory, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 711.

Grant and Bucklin (**6**, **7**) have proposed an empirical method of extrapolating stress-rupture data which is based on an analysis of the data on a log-log plot of stress *versus* rupture time. These plots are assumed to consist of a series of straight lines. The discontinuities are then attributed to structural instabilities or changes in the mode of deformation or fracture. It is proposed that a linear relation exists between: (1) the log time at the instability points and the test temperature, and (2) the log of the slopes of the various straight line segments and the test temperature. If these relations are determined by short-time tests at high temperatures, then it is proposed that the isothermals at longer times and lower temperatures can be predicted by extrapolation of the above-mentioned plots. This method has required, in the case of the alloys reported, that data be obtained at extremely short rupture times (0.001 to 0.01 hr) in order to obtain a sufficient number of discontinuities for the analysis. The special nature of the data required by this method renders checking difficult without suitably designed experiments on a large number of materials.

In another empirical approach, Manson and Haferd (**4**) have proposed a correlation parameter in the form $\frac{T - T_a}{\log t - \log t_a}$ where T_a and $\log t_a$ are material constants. This parameter expresses the observed tendency of plots of log t *versus* T at a constant nominal stress to be linear in the rupture time range above 30 hr, and for such lines representing different stress levels to converge to a single point $[T_a, \log t_a]$. Examination of published stress-rupture data for a large number of materials indicated this parameter would correlate data from 10 hr to as high as experimentally determined (in some cases as high as 10,000 hr). The determination of the material constants T_a and $\log t_a$ involved a certain amount of cross-plotting to obtain constant nominal stress curves, and it was recommended that the direct experimental determination of constant nominal stress data was to be desired for the determination of these constants. The number of data points required for this purpose was left to experimental investigation.

Since the complicated mechanism of creep and stress rupture is not completely understood, the viewpoint of the present report is that the data should be examined for experimentally consistent relations between the variables, which will achieve the practical objective of permitting the use of moderate-time data to predict the long-time data. To be useful, the relations must be sufficiently general so that establishment of their validity on materials for which a large amount of data are available will permit their application with confidence to materials of limited available data.

In the present report experimentally determined curves of constant nominal stress are presented for five materials. The data are analyzed on the basis of the linear parameter, the Larson and Miller parameter with $C = -20$ and for optimized values of C, and on the basis of a more general method described in the report. Emphasis is placed on the conformity of shape between the predicted and experimental curves in the experimental rupture time range as a guide to the suitability of extrapolation. The feasibility of representing stress, temperature, and rupture time by an explicit formula is also investigated.

TABLE I.—COMPOSITION, HEAT TREATMENT, AND HARDNESS OF ALLOYS TESTED.

Alloy	Composition, per cent								Heat Treatment	Rockwell Hardness
	Carbon	Chromium	Nickel	Molybdenum	Manganese	Silicon	Iron	Others		
DM	0.12	1.27		0.52	0.44	0.75	Balance		1550 F 1 hr Furnace	$R_B = 81$
"17-22A" S	0.30	1.25	0.25	0.52	0.63	0.60	Balance	V—0.25	1725 F, ½ hr, Air 1200 F, 6 hr, Air	$R_C = 33$
16-25-6 M	0.07	16.16	15.60	6.10	7.10	0.42	Balance		Water quench from 2150 F	...
16-13-3	0.11	16.80	11.66	2.18	1.59	0.70	Balance		Water quench from 2000 F	$R_B = 80$
C 422	0.23	13.19	0.65	1.03	0.81	0.16	Balance	V—0.25 W—0.84	1900 F, ½ hr, Oil 1200 F, 2 hr, Air	$R_C = 33$

MATERIAL AND PROCEDURE

The materials investigated and their chemical composition, heat treatment, and hardness are shown in Table I. The alloys were supplied as 1-in. hot-rolled bar stock. All specimens blanks for a given alloy were from the same heat and were heat treated in one group.

The specimen employed was identical to that used by Brown, Jones, and Newman (**8**) in the investigation of the smooth bar properties of various high-temperature alloys. The testing technique has been adequately described elsewhere (**8**, **9**). For this investigation, several different stress levels[3] were selected for each alloy covering the range of expected service application. At each of these stress levels, the temperature was varied to obtain points at various times to rupture. In general, it was attempted to establish carefully the behavior at a given constant nominal stress between times of 0.1 and 1000 hr. The experimental results are shown in Fig. 1. To improve the clarity of comparison between the experimental and analytical results no average curves have been drawn to represent the experimental data.

ANALYSIS

The approach in interpreting the results will be to compare the experimentally determined constant nominal stress curves to the corresponding curves calculated on the basis of several methods. In each of the methods to be examined it is inherent that the calculated curve coincide with the experimental curve for at least one point owing to the very nature of the construction of the "master" curve for the correlation. Hence, the placement of the calculated curve in the vicinity of the experimental points is not a significant indication of

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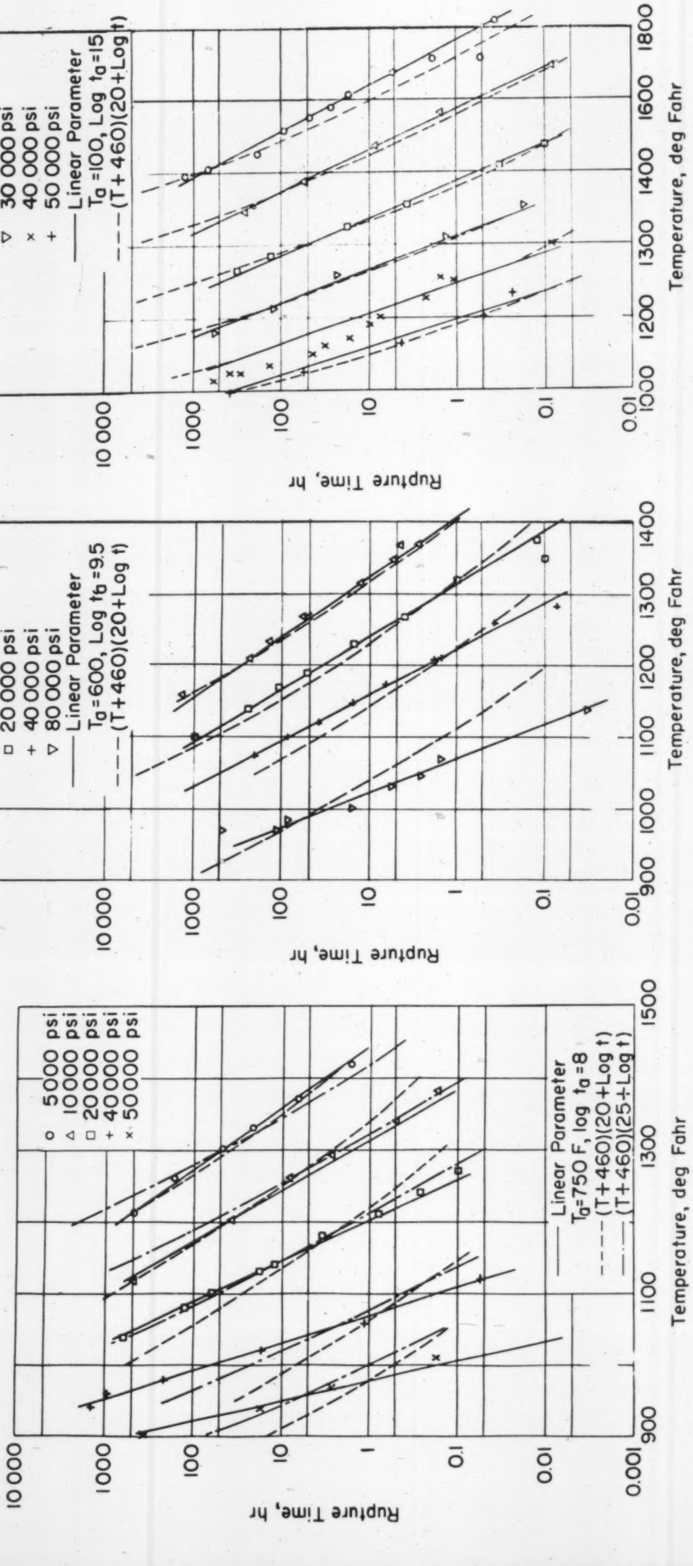
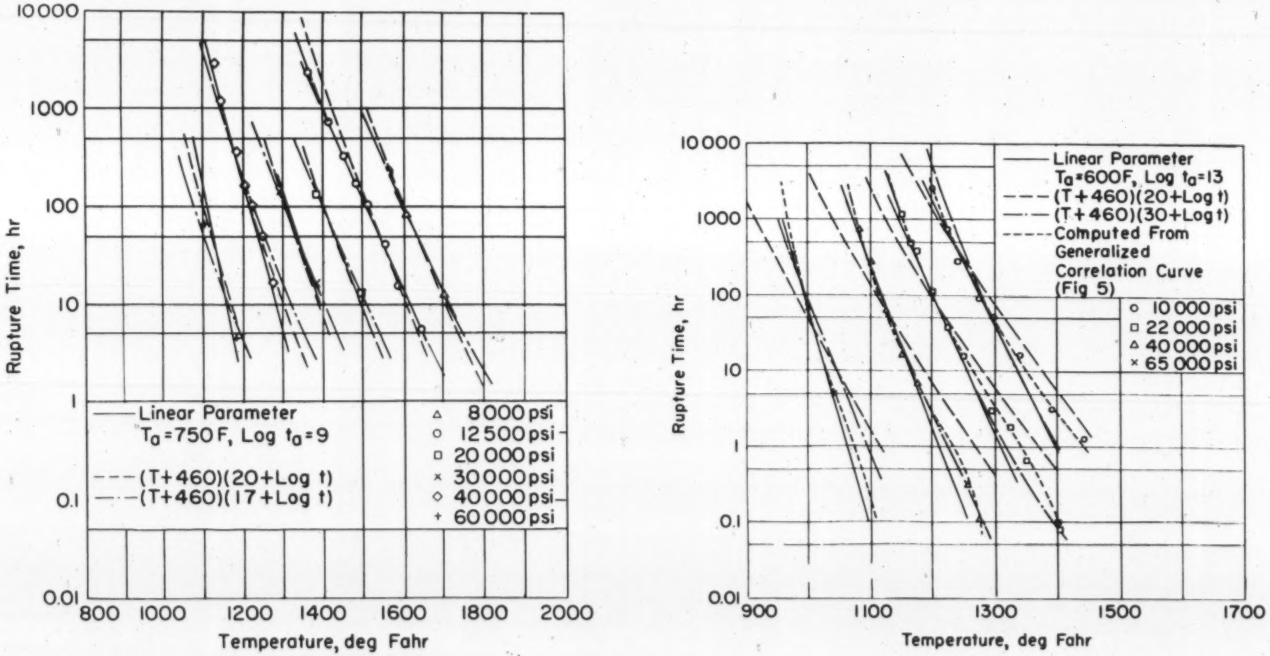
[3] These tests have been referred to as constant nominal stress tests since the load is maintained constant to fracture.



(a) DM steel.

(b) "17-22A"-S.

(c) 16-13-3.



(*d*) 16-25-6M.

(*e*) Crucible 422.

FIG. 1.—Constant Nominal Stress Data and Comparison of Various Correlating Parameters.

the success of the parameter to correlate the data. The more important criterion is the degree of coincidence between the calculated and experimental results over the entire range of time, temperature, and stress examined, since deviations in the experimental range are, in general, exaggerated by extrapolation. A further criterion is the applicability of a given method to several materials.

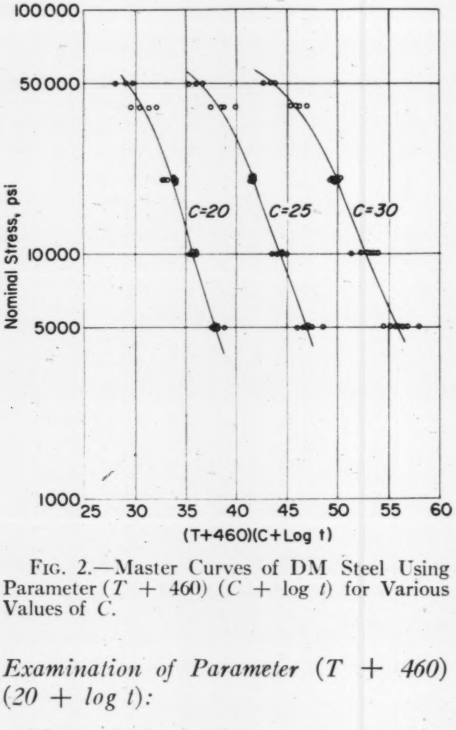


FIG. 2.—Master Curves of DM Steel Using Parameter $(T + 460)$ $(C + \log t)$ for Various Values of C.

Examination of Parameter $(T + 460)$ $(20 + \log t)$*:*

Figure 2 shows the master curve for DM steel to represent the type of difficulties sometimes encountered with this type of analysis. Considerable spread may exist among the points at each stress level, and some judgment must be exercised in fairing a mean master curve through the points. By favoring the points representing the longer rupture times, agreement can be achieved between calculated and experimental constant nominal stress curves in this time range at the expense of poor agreement in the lower rupture time range. Conversely, agreement in the lower time range can be achieved only at the expense of poor correlation of the higher rupture times. In the present analysis all the data were used for establishment of the master curve for each material. From these master curves the calculated constant nominal stress curves were constructed at each of the stress levels experimentally investigated. The results are shown as the dotted lines in Fig. 1. For DM steel, Fig. 1 (*a*), agreement between the experimental points and the calculated curve at a given constant nominal stress is satisfactory only in a very limited range of time and temperature, and deviations become very appreciable beyond this range. As an extreme example, at 900 F the master curve would yield a rupture time of 15 hr, whereas the experimental rupture time is 400 hr. At 40,000 psi and 960 F the calculated rupture time is 25 hr, while the experimental value is 930 hr. In most ranges the deviations are, of course, smaller; however, the lack of conformity of general shape between the calculated curves and experimental points would indicate the questionability of the use of this parameter for extrapolation beyond the experimental rupture time range.

For "17-22A" S steel, Fig. 1(*b*), the parameter $(T + 460)(20 + \log t)$ correlates the data for 10,000 psi very well. Deviations and discrepancy in curve shape are evident as the stress is increased. The greatest discrepancies would be expected at 80,000 psi; unfortunately, however, the data at this stress level exhibit the greatest scatter encountered in the investigation and therefore may not be conclusive.

For 16-13-3 steel, Fig. 1(*c*), only small deviations is seen to exist between the calculations based on the $(T + 460)$

(20 + log t) parameter and the 10,000, 20,000, and 30,000-psi curves. The slight curvature of the calculated lines may indicate the computed life at times longer than investigated would be too high. At 5000 psi the discrepancy in shape is detectable, but not large. At 40,000 psi unusual changes in curvature were detected not correlatable by the $(T + 460)(20 + \log t)$ parameter (or, as will later be seen, by any of the parameters examined). The large number of points used to establish the curve at this stress level would seem to indicate that the curvature is real and not the result of experimental scatter. The cause of this curvature and the inconsistency of this curve with those at other stress levels is not known. It should be pointed out that the reason for the coincidence of the theoretical curve with the experimental data in the 1-hr rupture time range, despite the preponderance of experimental data at the higher rupture times, is the requirement imposed by the authors of maintaining a smooth master curve.

For 16-25-6 M, Fig. 1(*d*), only two stress levels were investigated over a wide time range. At 40,000 psi the computed curve agrees very well up to 3000 hr, which was the highest time investigated for this stress level; but at 12,500 psi the calculated curve is somewhat too steep. The other stress levels were examined at two closely spaced temperatures, and it is therefore to be expected that near agreement between the computed curve and experimental points would exist.

In general, rather poor agreement between the curves calculated on the basis of $(T + 460)(20 + \log t)$ and the experimental points at four stress levels is seen for Crucible 422 steel in Fig. 1(*e*). While there is fairly good agreement at rupture times between 50 and 150 hr, considerable deviations are observed at higher and lower rupture times. Within the experimental time range this discrepancy is a factor of 3 or more. Thus, it would be expected that extrapolation of the data by the use of this parameter would result in considerable error.

Examination of Parameter $(T + 460)(C + \log t)$*:*

Attempts were made to find improved values of C for use in the parameter $(T + 460)(C + \log t)$ as follows: Plots were first made of log t *versus* $\dfrac{1}{T + 460}$ at constant nominal stress to ascertain whether a single point of convergence could be found on the log t axis. Since convergence was never strictly characteristic, a number of values of C were selected to cover the range of extrapolated intercepts. Plots were then made of stress *versus* $(T + 460)(C + \log t)$ and a mean master curve was constructed for each of the several values of C. Based on the mean master curve, computed constant nominal stress curves were constructed and compared to the experimental points. The value of C that produced best average coincidence among all the stress levels was selected as the optimum value.

The general effects of variation of C on the master curves is illustrated in Fig. 2 by the example of DM steel. It is noted that the lowest value of C tends to minimize the spread of points at the lower stresses, while increasing the value of C tends to reduce the spread at the higher stress levels but at the expense of the correlation at lower stresses. This effect of C was noted to a greater or less extent depending on the alloy, and when it was large, as in the case of DM steel, the selection of an optimum value was difficult.

The calculated constant nominal stress curves for the optimum values of C are shown by the dot-dash curves for each of the five materials in Fig. 1. For DM steel, considerable discrepancy still exists between the shapes of the computed curves and the experimental points, as shown in Fig. 1(*a*). The selection of higher values of C would cause all the dot-dash curves to become steeper, thereby improving the shape of the curves at the higher stress levels, but the discrepancies at the lower stress levels would be aggravated. On the other hand, selection of lower values of C would make all the curves less steep, thus improving the shape of the lower stress levels at the expense of fit of the higher stress levels. These trends reflect the spread of points on the master curves of Fig. 2.

For "17-22A" S steel the value $C = 20$ was found to be optimum. The value of $C = 20$ is likewise selected as the optimum for 16-13-3 steel, Fig. 1(*c*), since as already discussed the value correlated the data quite well except at the stress of 40,000 psi.

For 16-25-6 M steel (Fig. 1(*d*)) the value $C = 17$ improves the correlation at 12,500 psi with only a slight penalty on the correlation at 40,000 psi; hence $C = 17$ is considered as the optimum value for this material. For Crucible 422 steel the value $C = 30$ shows very great improvement over the correlation using $C = 20$, although at 10,000 psi the computed curve is too steep.

Examination of the Parameter

$$\frac{T - T_a}{\log t - \log t_a}.$$

For correlation of the data on the basis of the linear parameter, the constant nominal stress curves are approximated by straight lines converging to a single point $[T_a, \log t_a]$.

Manson and Haferd (**4**) recommended that the point of convergence be located by use of data in the 30 to 300 hr rupture time range for two reasons. First, on the basis of analysis of the published isothermal data it appeared that curvature existed in the constant nominal stress curves for times less than 30 hr. Second, it was desired to make use of this parameter for extrapolation of data obtained in the range of moderate rupture times (that is, less than 300 hr).

To obtain the values T_a and $\log t_a$ each set of constant nominal stress data was first faired by a curve using all the available data below 300 hr. Tangets to these curves in the 30 to 300-hr time range that converged to a single point then established the point T_a, $\log T_a$. For all the materials except Crucible 422 the data below 30 hr were nearly coincident with the tangents in the 30 to 300-hr time range.

Construction of constant nominal stress curves on the basis of the linear parameter can be achieved either by passing straight lines through the established point (T_a, $\log t_a$) and the mean of the data points in a selected rupture time range, or by constructing a master curve of $\log \sigma$ *versus* $\dfrac{T - T_a}{\log t - \log t_a}$, and reconstructing the straight lines at each stress level. The two methods are essentially equivalent, since the fairing of the master curve establishes the best value of $\dfrac{T - T_a}{\log t - \log t_a}$ for each stress level.

It will be seen from Fig. 1(*a*) to 1(*d*) that the calculated lines agree very well with the experimental data for DM, "17-22A" S, 16-13-3, and 16-25-6 M steels over a very wide range of time, temperature, and stress. In most cases the trend of the data can be approximated by straight lines to rupture times of 0.1 hr and as high as was experimentally investigated (1000 to 3000 hr, depending on the material). The only distinct anomaly is the 40,000-psi curve for 16-13-3 steel. For Crucible 422 the linear parameter characterizes the data well in the experimental time range above 30 hr, but the trend of the short-time data indicates that the constant nominal stress lines possess curvature. Because of this curvature and the recognition of the fact that other alloys not yet investigated in this manner may also possess some arbitrary curvature, there appears to be a need for a correlation procedure capable of handling materials exhibiting such behavior.

General Approach to the Correlation of Stress-Rupture Data:

A more general approach to the problem of the correlation of stress-rupture data will be presented by illustrating treatment of the Crucible 422 data. It will be recognized that this method will produce results identical with those obtained by the linear time-temperature parameter, if the constant nominal stress curves are linear. Therefore, the detailed analysis of the four remaining materials will not be presented.

For the purposes of a general correlation of the data it will be assumed that a correlation of rupture time, temperature, and stress can be achieved in the form of a product of functions of each of the variables. Thus the correlation will be in the form

$$F(t) \cdot G(T) = P(\sigma) \qquad \text{..........(1)}$$

where:

t = rupture time,
T = temperature, deg Fahr,
σ = stress, psi, and
F, G, and P = functions to be determined.

This assumption is consistent with the Larson-Miller parameter in which $F(t) \equiv C + \log t$, $G(T) \equiv T + 460$, and $P(\sigma)$ is expressed graphically as the master curve. It is consistent with the linear parameter in which $F(t) \equiv (\log t - \log t_a)^{-1}$, $G(T) \equiv T - T_a$, and $P(\sigma)$ is again expressed graphically as a master curve. It is not consistent with the Machlin-Nowick rate process equation in which a stress term appears as an addition to a temperature term, and it would not be consistent with the $(T + 460)(C + \log t)$ parameter if C were a function of stress rather than being a material or universal constant. Thus it must be recognized that although Eq 1 is quite general, there is a possibility that it is not sufficiently general to permit separation of the variables into product terms.

Corresponding-Temperature Plot:

For the purpose of generalizing the results, it is convenient to introduce the concept of a corresponding-temperature plot. This plot was first used to ascertain the possibility of correlating stress-rupture data on the basis of the Larson and Miller parameter $(T + 460)(C + \log t)$ where C is a constant other than 20. Let it be assumed, for example, that a suitable value of C can be found for perfect correlation of the data for Crucible 422 steel. Then the equation of the 22,000 psi constant stress line would be

$$(T_{22,000} + 460)(C + \log t_{22,000}) = P_{22,000} \qquad (2)$$

and that for the 10,000 psi line would be

$$(T_{10,000} + 460)(C + \log t_{10,000}) = P_{10,000} \qquad (3)$$

Now select identical values of rupture time on each of the two curves and divide Eq 2 by Eq 3. Since C is the same in each equation and since $\log t$ is the same by selection, the term $(C + \log t)$ cancels upon dividing and the relation obtained becomes

$$\frac{T_{20,000} + 460}{T_{10,000} + 460} = \frac{P_{22,000}}{P_{10,000}} = \text{constant} \qquad \text{...(4)}$$

Thus a plot of $T_{22,000}$ against $T_{10,000}$ at arbitrary selected identical values of rupture time should, by Eq 4, yield a straight line intersecting the 45-deg line at the point $T_{22,000} = T_{10,000} = -460$. Similarly using the 22,000 psi as the base curve, plotting corresponding temperatures for identical rupture times for each of the other stress curves in Fig. 1(*e*) against the temperature on the 22,000 psi curve should yield a series of straight lines all converging to the point −460 F on the 45-deg line. The corresponding-temperature plot for Crucible 422 steel is shown in Fig. 3(*a*). It is seen that the lines are quite straight, but their apparent point of convergence on the 45-deg line is at 700 rather than at −460 F. The compromise that must be made if the temperature term in the parameter is to be taken as $(T + 460)$ is shown by the dotted lines which converge to the point −460 on the 45-deg line. Thus, on the basis of the corresponding-temperature plot, it can be concluded that, although a better value of C than 20 might be found for correlation purposes, some error must in any case be accepted because of the use of absolute temperature as the multiplier for the $(C + \log t)$ term.

Figure 3(*b*) shows the corresponding-temperature plot for DM steel on which more data were obtained than on Crucible 422 steel and for which the point of convergence is more distinctly defined as a value in the vicinity of 750 F rather than −460 F. The corresponding-temperature plot thus serves the purpose of assisting in the identification of suitable forms for the temperature characteristic $G(T)$.

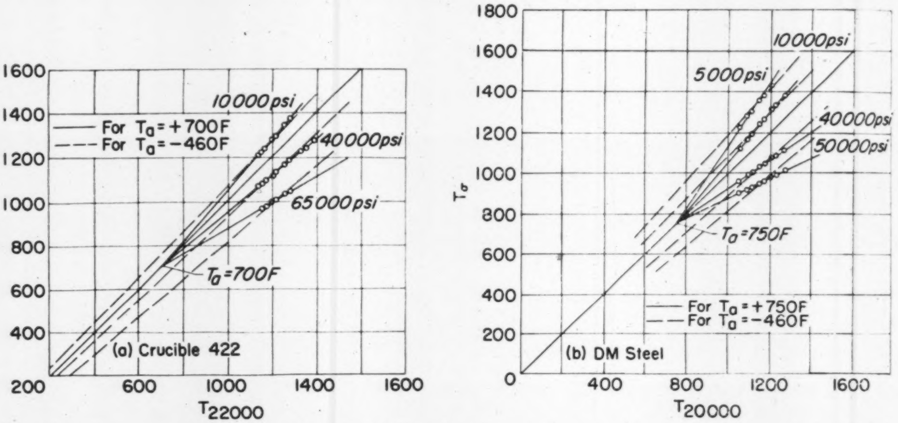


FIG. 3.—Corresponding-Temperature Plot Showing Convergence to $T_a = +700$ F and the Result of Assuming $T_a = -460$ F.

The Temperature Characteristic:

Consider two constant stress curves σ_1 and σ_0. Then by Eq 1 the equations of these curves are

$$F(t_1) \cdot G(T_1) = P(\sigma_1) \qquad (5)$$

$$F(t_0) \cdot G(T_0) = P(\sigma_0) \qquad (6)$$

Using the concept of the corresponding-temperature plot select identical rupture time values on each of the two curves and plot the corresponding temperatures T_1 against T_0. The equation of the relation between T_1 and T_0 is obtained by dividing Eq 5 by Eq 6, thereby cancelling the time function which is identical for all selected values:

$$\frac{G(T_1)}{G(T_0)} = \frac{P(\sigma_1)}{P(\sigma_0)} = Q_1 \text{, a constant} \qquad (7)$$

When such plots are made using the various stress curves in combinations, the corresponding temperatures lie on very nearly straight lines which converge to a point T_a on the 45-deg line. This representation should obviously yield the same value for T_a as obtained using the linear time-temperature parameter provided the constant nominal stress curves are linear and converge to the point T_a, log t_a. However, the property of linearity and convergence of the corresponding temperature plots was found to be characteristic for Crucible 422 where curvature exists in the constant stress curves.

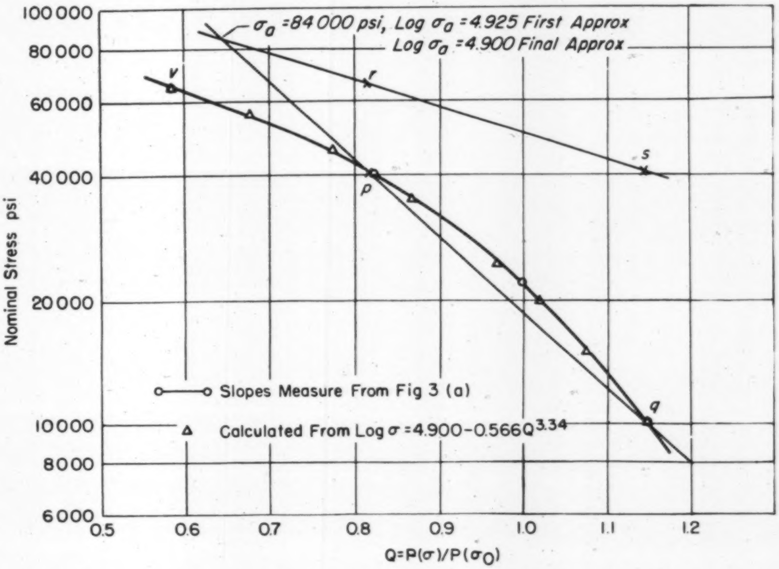


FIG. 4.—Relation Between Q and Nominal Stress for Crucible 422 Determined from Corresponding-Temperature Plots and the Results Calculated from an Equation Derived to Fit This Relation.

It can be seen either by inspection or by setting up a suitable differential equation expressing the fact that Eq 7 must define a straight-line relation between T_1 and T_0 which is satisfied at a common point on the 45-deg line for all assigned constant values of Q, that a general form for the function G is found to be $(T - T_a)^z$. For this form, Eq 7 becomes

$$\left(\frac{T_1 - T_a}{T_0 - T_a}\right)^z = \text{constant} \quad \ldots\ldots (7a)$$

which establishes the necessary linear convergent relation when the zth root is taken of both sides of the equation. At this point of the analysis, z is completely arbitrary, since substitution for G in Eq 1 can be followed by taking zth roots of both sides of this equation without destroying the generality of F and P which have not as yet been established. The simplest temperature characteristics result when z is taken as unity. Hence, without destroying the generality of the analysis

$$G(T) \equiv T - T_a \ldots\ldots\ldots\ldots (8)$$

where T_a is the apparent point of convergence on the 45-deg lines of the corresponding-temperature plots. In some cases, particularly if there is scatter in the basic data, the convergence of corresponding-temperature plots to a point T_a may not be as distinct as in the case of Fig. 3. However, the arbitrary selection of an approximate point that best satisfies the linearity and convergence property is usually found to give good results if consistency is maintained in the subsequent analysis. The value of T_a is not critical, and good correlation can be obtained for a fairly wide range of selected values for a given material.

A point of practical significance in the construction of the corresponding-temperature plot is the proper selection of the constant nominal stress curve used as a base. It will be recognized that an experimental scatter in the base curve will be reflected in each of the corresponding-temperature lines. Since it may in certain cases be difficult to ascertain which one of the constant stress curves is most suitable as a base, it would be desirable to establish a reference curve on the basis of all the available data. This can be done as follows. The numerical average of the temperatures on each of the available constant stress curves is found for an arbitrary time level. The temperature so obtained can be shown to correspond to that on some constant nominal stress curve. This curve may then be defined as completely as desired by selecting another arbitrary time level, again averaging the temperatures. The average curve so obtained should then be the most suitable base for fairing the data.

It should also be pointed out that although from the standpoint of correlation of stress-rupture data the selection of $G(t)$ as $(T - T_a)$ is adequate, there may be other forms of $G(T)$ which characterize linearity and convergence of corresponding-temperature plots as an approximate property. Such forms would not be derivable from the differential equation which expresses this as an exact property. For example, the form $G(T)$ $\equiv \frac{1}{T + 460} - \frac{1}{T_a + 460}$, which might be obtained from the rate process equation if plots of log t *versus* $\frac{1}{T + 460}$ were linear but converged to a point at absolute temperature $T_a + 460$, would show some tendency to produce approximately linear and convergent corresponding-temperature plots in a limited temperature range. This function is usually inferior for representing the experimental data; however, it is conceivable that there may be forms of functions derivable from physical theory and consistent with the observed properties of corresponding-temperature plots. If the correlation method is to provide insight into the fundamental mechanism of the stress-rupture process, the temperature characteristic may have to be re-examined.

Stress Characteristic:

The corresponding-temperature plot also provides considerable information about the stress characteristic. Substituting Eq 8 into Eq 7 gives

$$\frac{T_1 - T_a}{T_0 - T_a} = \frac{P(\sigma_1)}{P(\sigma_0)} = Q_1 \dots\dots\dots (9)$$

Since $\frac{T_1 - T_a}{T_0 - T_a}$ is the slope of line associated with T_1 on the corresponding-temperature plot, the value of Q_1 may be determined. Similarly the values of $Q_2 = \left(\frac{P(\sigma_2)}{P(\sigma_0)}\right), Q_3 = \left(\frac{P(\sigma_3)}{P(\sigma_0)}\right)$, etc., for each of the experimentally available constant stress curves can be determined. The Q function can thus be plotted against stress. Figure 4 shows the values for Crucible 422 steel based on the corresponding-temperature plot of Fig. 3(*a*) in which the 22,000-psi constant nominal stress curve was used as the baseline.

The Time Characteristic:

Once $G(T)$ and Q have been determined as described above, the time characteristic can be obtained from Eq 1. Because experience has indicated that $\dfrac{T - T_a}{\log t - \log t_a}$ is suitable in most cases for correlation and since $G(T)$ has already been determined as $T - T_a$ it will be convenient to solve Eq 1 for $\dfrac{1}{F(t)}$ rather than $F(t)$. Thus,

$$\frac{1}{F(t)} = \frac{G(T)}{P(\sigma)} = \frac{T - T_a}{P(\sigma)}$$

$$= \frac{1}{P(\sigma_0)} \cdot \frac{T - T_a}{\dfrac{P(\sigma)}{P(\sigma_0)}} = \frac{1}{P(\sigma_0)} \cdot \frac{T - T_a}{Q} \quad \ldots (10)$$

Hence a plot of T *versus* $\dfrac{T - T_a}{Q}$ should yield a single curve for all the data which will define $F(t)$.

For Crucible 422 a plot of the time characteristic is shown in Fig. 5 where $\log t$ has been plotted *versus* $\dfrac{T - T_a}{Q}$ using the values of Q plotted in Fig. 4. It is evident that the data correlate very well. The change in curvature in the time range below one hour is not well established by the few available data points. The curve has, however, arbitrarily been drawn to pass through as many of the data points as possible, necessitating the introduction of a change of curvature. It is evident that the time characteristic obtained in this manner, can be made to conform as closely as possible to the original data points and does not necessitate that the characteristic have any pre-established shape, such as straight lines in cases where the linear parameter is valid and hyperbolas where the $(T + 460)(C + \log t)$ parameter is valid.

The individual constant nominal stress curves can be reconstructed from the time characteristic in Fig. 5 and the Q value for each stress level as shown in Fig. 4. The short-dashed curves in Fig. 1(*e*) were constructed in this manner for each of the experimentally investigated stress levels for Crucible 422. It will be seen that the curves so obtained agree very well with the data over the entire experimental range.

Since the time characteristic can be extended up to the longest rupture time experimentally available, regardless of the particular stress level at which the data point was obtained, the curve essentially serves the purpose of permitting extrapolation for those stress levels where only lower rupture times are experimentally available. In addition, a certain amount of extrapolation of the time characteristic curve should be possible, thereby extending the experimental time range with a degree of confidence. Thus, in Fig. 5, in the absence of more suitable methods of extrapolation, the last portion of the time characteristic could probably be extended linearly up to about 10,000 hr.

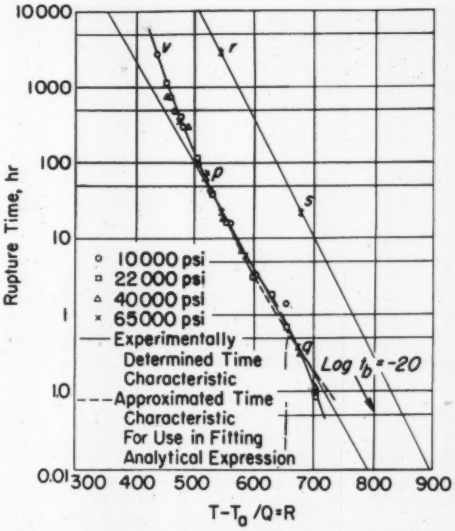


FIG. 5.—Experimentally Determined Time Characteristic for Crucible 422.

The results would, of course, be slightly different from those obtained on the basis of the linear time-temperature parameter when only the 30 to 300 hr data are used for the determination of T_a and $\log t_a$. The latter case corresponds to the linear extension of the tangent to the time characteristic curve in the 30 to 300 hr range.

When the individual constant nominal stress curves are linear, the time characteristic will also be linear, intersecting the vertical axes at a time corresponding to $\log t_a$.

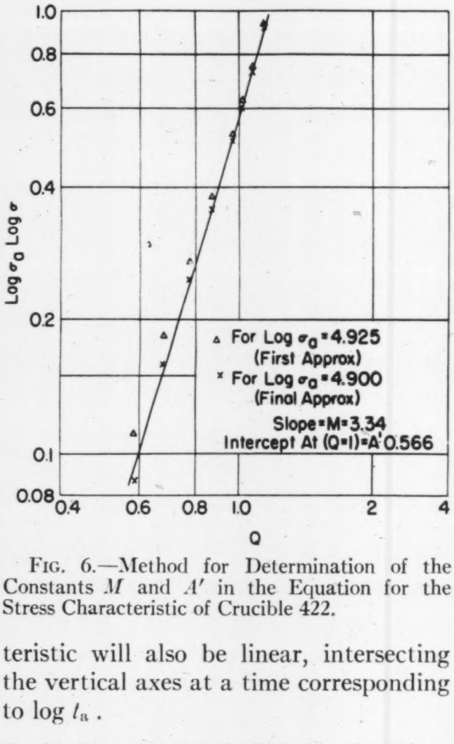


FIG. 6.—Method for Determination of the Constants M and A' in the Equation for the Stress Characteristic of Crucible 422.

Derivation of a Single Relation for Time, Temperature, and Stress:

It has been shown in Figs. 4 and 5 that the characteristics of stress and time can be defined graphically. It is possible to fit an analytical expression to each of the curves and by combining these with the temperature function already determined, to write an explicit analytical expression in terms of time, temperature, and stress.

If Crucible 422 is taken as an example the stress function in Fig. 4 can be taken analogous to the temperature characteristic:

$$Q = A(\log \sigma_a - \log \sigma)^m \ldots\ldots (11)$$

or

$$\log \sigma = \log \sigma_a - A'Q^M \ldots\ldots (12)$$

The curve in Fig. 4 may be fitted to this equation by a method outlined by Running (**10**). The point p is selected on the curve with an abscissa value equal to the mean proportional between the abscissa values of the end points v and q. The point r is selected to have the same abscissa as p and the same ordinate as v, while the point s is selected to have the same abscissa as q and the same ordinate as p. The intersection of the lines drawn through r and s, p and q give a value for the first approximation to σ_a, in this case equal to 84,000 psi. Since Eq 12 can be rewritten in the following form:

$$\log (\log \sigma_a - \log \sigma) = (\log A' + M \log Q) . (13)$$

a plot of $\log (\log \sigma_a - \log \sigma)$ *versus* $\log Q$ should be linear with a slope equal to M and an intercept at $\log Q = 1$ of $\log A'$. The linearity of this plot is a test for the suitability of the selected value of σ_a by the method outlined above. Such a plot is shown in Fig. 6. The results for $\sigma_a = 84{,}000$ ($\log \sigma_a = 4.925$) exhibit some deviation from linearity. The arbitrary selection of $\log \sigma_a = 4.900$ improved the linearity and this value was therefore chosen. The values of M and A' are shown in Fig. 6, and these values can now be substituted in Eq 13 to yield the following expression:

$$\log \sigma = 4.900 - 0.566\, Q^{3.34} \ldots\ldots (14)$$

or

$$Q = 1.186\ (4.900 - \log \sigma)^{0.3} \ldots\ldots (15)$$

The results calculated from the expression agree well with the curve as shown in Fig. 4.

For the time characteristic represented in Fig. 5, the form by analogy with the temperature and stress characteristic and for consistency with the linear parameter may be taken as

$$R = B(\log t - \log t_b)^n \quad \ldots\ldots (16)$$

or

$$\log t = \log t_b + B'R^N \quad \ldots\ldots (17)$$

The expression in Eq 17 will not fit the curve drawn through the experimental points in Fig. 5 because of the previously discussed slight change in curvature at times around 1 hr. To provide a smooth curve for the purpose of fitting, the dotted line has been taken to represent all the data. This curve may now be fitted by the same procedure as outlined for the stress characteristic. The value of $\log t_b = -20$ is large and not too critical. The values of B' and N' were determined from the plot in Fig. 7. The equation so obtained for the time characteristic becomes:

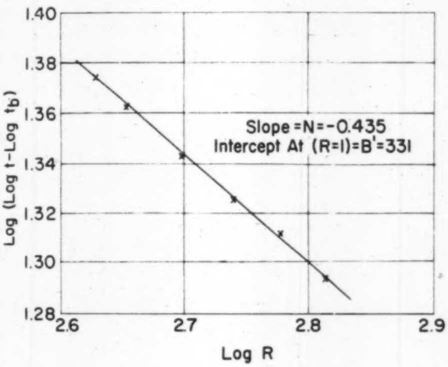


FIG. 7.—Method for Determination of the Constants N and B' in the Equation for the Time Characteristic for Crucible 422.

$$\log t = 331\, R^{-0.435} - 20 \quad \ldots\ldots (18)$$

or

$$R = \frac{T - T_a}{Q} = \frac{6.20 \cdot 10^5}{(20 + \log t)^{2.30}} \quad \ldots (19)$$

By combining Eq 15 with Eq 19 the following explicit expression is obtained in terms of time, temperature, and stress:

$$(T - 700)(20 + \log t)^{2.30} = 7.4 \cdot 10^5 (4.900 - \log \sigma)^{0.30} \quad \ldots (20)$$

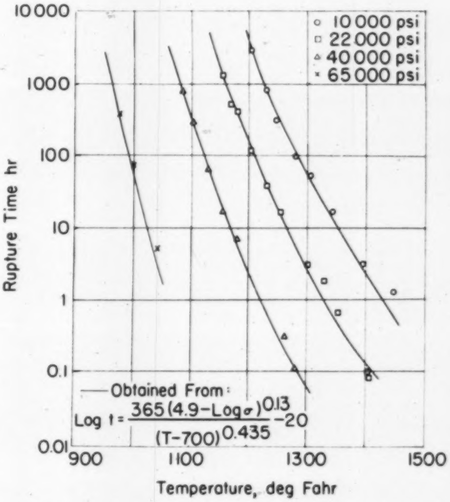


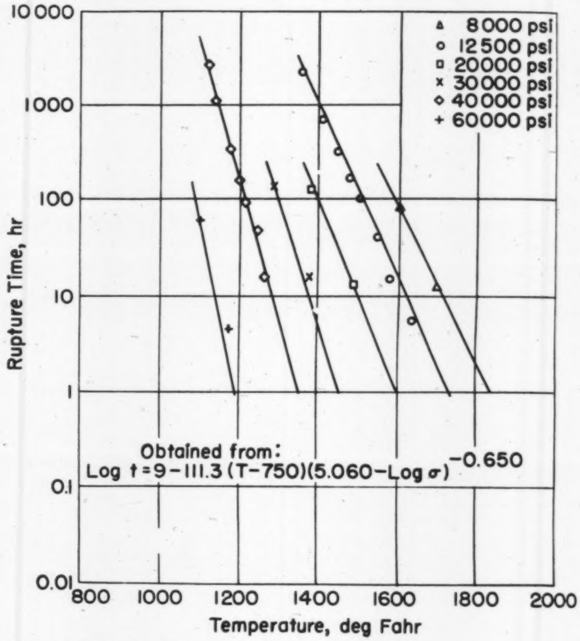
FIG. 8.—Comparison Between Experimental Data for Crucible 422 and the Curves Obtained from the Explicit Formula Relating Time, Temperature, and Stress.

or

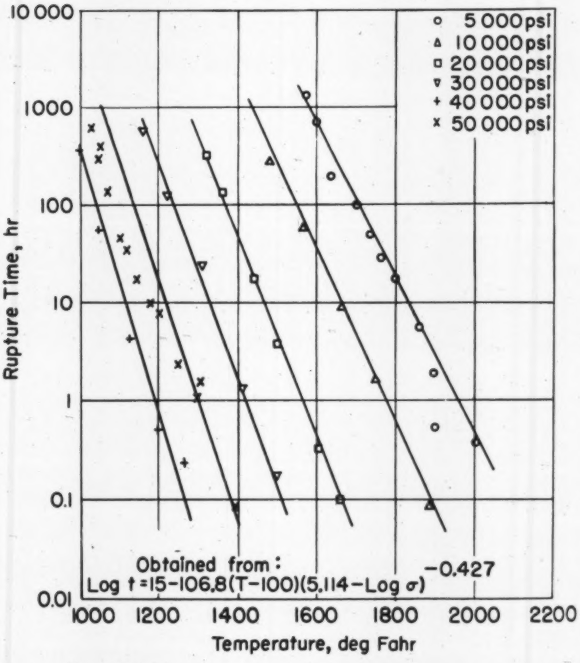
$$\log t = 356 \frac{(4.900 - \log \sigma)^{0.130}}{(T - 700)^{0.435}} - 20 \quad \ldots (21)$$

Using this expression, the constant nominal stress curves at each of the four experimentally investigated stress levels were calculated and are shown in Fig. 8. It is evident that these curves represent the data very well and therefore demonstrate the suitability of the form of Eq 20.

Single equations relating stress, temperature, and time to rupture have also been obtained for the other four materials investigated. In these cases, where the linear parameter

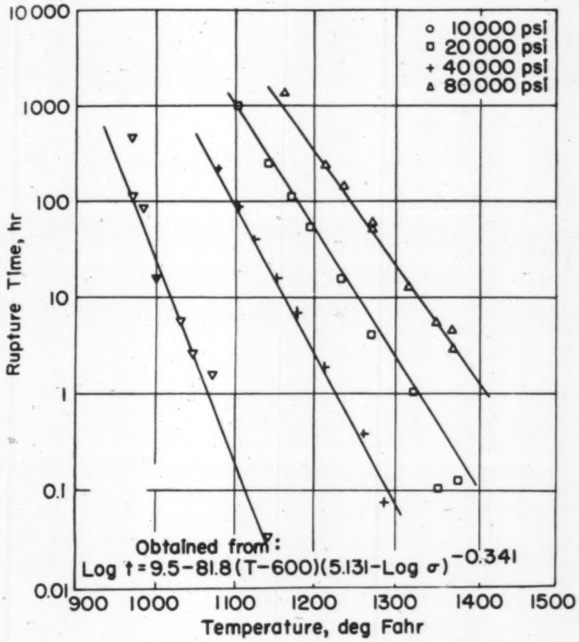


(*a*) 16-25-6M

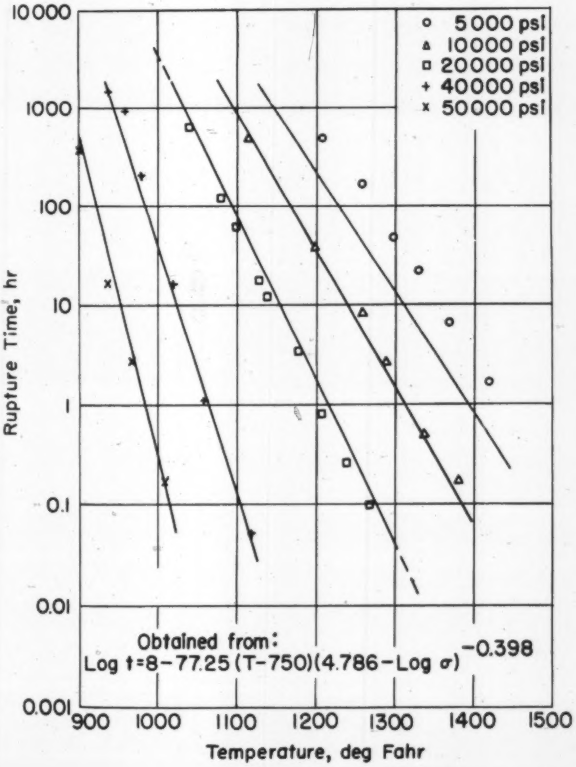


(*c*) 16-13-3

FIG. 9.—Comparison Between Experimental Data and the Curves Cal-



(*b*) 17-22A(S)



(*d*) DM Steel

culated from Explicit Formula Relating Time, Temperature, and Stress.

$$\frac{T - T_a}{\log t - \log t_a}$$

was suitable for characterizing the data in the entire experimental range, it was necessary merely to fit an equation of form (11) to the master curve, where Q in this case is replaced by the linear parameter. No difficulty was encountered in this process except for DM steel in which the master curve showed a reversal of curvature at stresses below 10,000 psi (similar to the reversals in the master curves shown by Manson and Haferd (**4**), especially for Inconel X). For DM, therefore, the curve was fitted only for stresses above 10,000 psi, and it was expected that poor agreement between the formula and the experimental data would occur at 5000 psi. The equations deduced for each of the four materials are as follows:

$$\left.\begin{aligned}
&\text{16-25-6M:}\quad \frac{T - 750}{\log t - 9}\\
&\qquad = -111.3(5.060 - \log \sigma)^{-0.650}\\
&\text{“17-22 A” S:}\quad \frac{T - 600}{\log t - 9.5}\\
&\qquad = -81.8(5.131 - \log \sigma)^{-0.341}\\
&\text{16-13-3:}\quad \frac{T - 100}{\log t - 15}\\
&\qquad = -106.8(5.114 - \log \sigma)^{-0.427}\\
&\text{DM:}\quad \frac{T - 750}{\log t - 8}\\
&\qquad = -77.25(4.786 - \log \sigma)^{-0.398}
\end{aligned}\right\}\ldots(22)$$

Figure 9(*a* to *d*) shows the comparison between the experimental data and the computed results based on Eq 22. In general, the agreement is seen to be very good except for the 5000 psi curve for DM steel, the reason for which was previously described. Thus it can be seen that over a fairly wide range of time, temperature, and stress, the variables can be related by an explicit expression in the form

$$(T - T_a)(\log t - \log t_a)^{\alpha} = A(\log \sigma_a - \log \sigma)^{\beta}$$

where in most cases $\alpha = -1$.

CONCLUDING REMARKS

Based on experimental determination of constant nominal stress curves on five materials and on the basis of attempts to correlate the results according to several different proposed methods, the following comments appear justified:

1. The use of the parameter $(T + 460)(20 + \log t)$ for correlation of data and for prediction of long rupture times from tests in the short- and medium-rupture time range may result in appreciable errors. The use of the parameter $(T + 460)(C + \log t)$ considerably improves the correlation in some cases. The data for DM steel, however, could not be correlated by this parameter for any single value of C. It is also difficult to ascertain the improved value of C unless a large amount of experimental data are available, in which case the data may be sufficient for analysis by the linear parameter method or by the more general method described in this paper.

2. Very good correlation was obtained on the basis of the linear time-temperature parameter. The more general method described in this report is completely equivalent to the method based on the linear parameter in such cases and in such regions where the log t *versus* T curves at constant nominal stress are linear. The need for the method arises in cases where curvature is observed in the plots at constant nominal stress. In these cases the regions with curvature can be used to assist in the determination of T_a and the stress characteristic, whereas in the linear parameter method no use can be made of these regions.

3. Linearity of plots of log t *verus* T persisted in rupture time regions in which

conventional isothermal plots show considerable curvature and where they would normally be characterized by "breaks" if plotted on log-log paper. Thus the presence of such "breaks" do not appear to be a deterrent in the feasibility of extrapolation from short and medium rupture time data. The limitations of the method have not, however, been fully established. It is, for example, desirable to investigate why some isolated constant nominal stress curves, such as 40,000 psi for 16-13-3 steel show unusual curvature in the time range when other constant stress curves are very nearly linear.

4. Representation of stress, temperature, and rupture time by an explicit expression in the form $(T - T_a)(\log t - \log t_a)^\alpha = A(\log\sigma - \log\sigma_a)^\beta$, where in most cases $\alpha = -1$ appears to give very good correlation except in some cases at very low stress levels. Such a relation may be quite useful when it is necessary to express one of the variables explicitly in terms of the others, or when it is necessary to perform the correlation from unsystematic data.

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DISCUSSION

Mr. J. D. Marble.[1]—We in the General Electric Co. have, of course, made considerable use of the Larson-Miller time-temperature parameter for stress-rupture data since its development two years ago and are, therefore, very pleased to see interest in such parameters expressed in the study made by the authors. They should be highly commended because the study is quite a good one and quite a complete one.

There are, however, a few points which should perhaps be brought out in comparing the NACA linear time-temperature parameter to the Larson-Miller time-temperature parameter. We do not hesitate to agree that application of the NACA parameter resulted in a much better correlation of the data than did the Larson-Miller parameter for those alloys studied in the paper. Perhaps the NACA parameter will prove significantly better for all alloys, in which case the simplicity advantage of the Larson-Miller parameter will no longer be so important. However, we feel this simplicity advantage should be clearly understood. Perhaps the most important point to us at this time is the usage of equipment. To use the NACA parameter efficiently, only small increments of temperature between tests at the same stress are possible in order to keep the times to failure in the 30 to 300-hr range suggested by Manson and Haferd in *NACA TN 2890*.[2] Therefore, maintaining many furnaces at different temperatures or changing temperature several times on a few furnaces can be very time consuming. This may not be quite as great a hardship for those laboratories with only single-station creep-rupture units, but most of the General Electric laboratories have multiple-station units for rupture evaluation work on which changes in temperature are tedious if proper temperature distribution is maintained. The Larson-Miller parameter is more attractive because 100 to 150 F intervals are all that are required. Another point of importance is the number of test points required to determine a curve for the material. Since the constant for the Larson-Miller parameter may be chosen to the nearest five units (that is, 15, 20, 25, 30) from experience with similar materials before the tests are made, only the stresses desired or times and temperatures desired need be covered during the testing. For example, perhaps only four specimens could be used to compare a development alloy with a commercial one over the expected service temperature and stress range or to compare a new heat of a particular material to those preceding. For the same test series, probably at least ten specimens would be required for the NACA parameter method since some would be required to establish the constants unless these could be assumed for this method in the same manner that they are for the Larson-Miller method. However, even so, more than one point at two or more stress levels would be required to check the constants. A third point of importance is the simplicity of application. With the Larson-Miller parameter, anyone can plot the data and produce the same curve as the test laboratory. However, with the NACA parameter the constants are determined by the plotter and are subject to wide variation because of the large amount of extrapolation required. Also, the constants may vary for different heats of the same material. For example, the authors tested two heats of DM steel and in *TN 2890* reported the constants as $T_a = 100$ F and log $t_a = 22$ for one heat and in the present paper reported the constants as $T_a = 750$ F and log $t_a = 8$ for the other heat. It would be difficult to compare these two heats on the same plot using the NACA parameter method.

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[1] Metallurgical Engineer, Evendale Plant Laboratory, Aircraft Gas Turbine Division, General Electric Co., Cincinnati, Ohio.

[2] S. S. Manson and A. M. Haferd, "A Linear Time-Temperature Relation for Extrapolation of Creep and Stress-Rupture Data," *NACA TN 2890*, NACA, Washington, D. C., March, 1953.

Variation in materials makes unnecessary the accuracy possible by the use of the NACA parameter method, except in a few important cases. Continuing the comparison of the two heats of DM steel reported by the authors, we find that no correlation method could have been used to predict the behavior of one heat from tests on the other because of the wide difference in properties. Yet this is one of the most common uses of rupture testing—that of predicting the behavior of subsequent heats of similar material from tests on one heat. Therefore, in such cases either an expected scatter band around the curve must be assumed or must be obtained from tests on numerous heats. A simple, common basis for comparing on the same plot several heats of the same material is required and seems to be offered by the Larson-Miller parameter. For the same type of predicting, it is sometimes necessary to know the rupture behavior of the material at temperatures or stresses intermediate between those tested but with only fair accuracy. The Larson-Miller parameter seems adequate here in nearly all cases, but the NACA parameter offers greater accuracy and probably should be used in cases where great accuracy seems necessary. But when tests are being made on a particular heat to determine the behavior of parts made from that particular heat, especially predicted long-time behavior such as in steam turbine materials, great accuracy of interpolation and extrapolation of data are required and the NACA parameter seems to supply this. We would recommend its use at least in these instances and possibly in many others when means of using it easily and with good repeatability have been developed. Here is a phase which we feel should be given some attention by the authors and others applying this method, this phase being that of easier application of the method. Things which should be done are making checks on predicting the constants for materials from similar previous materials, checks on the effect of errors in the determination of the constants, development and distribution of nomographs for easy determination of the parameter values from test results, and development of means of comparing at least similar materials on the same plot. These may all be simple, but their completion will greatly enhance the benefit to industry from this new parameter. Very few people are willing to accept a new method immediately, especially if at first glance it appears more complex than their present one.

We would recommend that all those interested in the authors' present paper also read the earlier treatise on this subject, *NACA TN 2890.*[2]

MR. JAMES MILLER.[3] (*by letter*).—The authors' commendable analysis gives new support to the idea that a parameter of time and temperature can be used to great advantage in the correlation and extrapolation of rupture data. The linear temperature parameter, which they indicate as having somewhat greater accuracy than the parameter $T(C + \log t)$, requires, however, sufficient data for the evaluation of two material constants.

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[3] Thomson Laboratory, Metallurgy Section, General Electric Co., West Lynn, Mass.

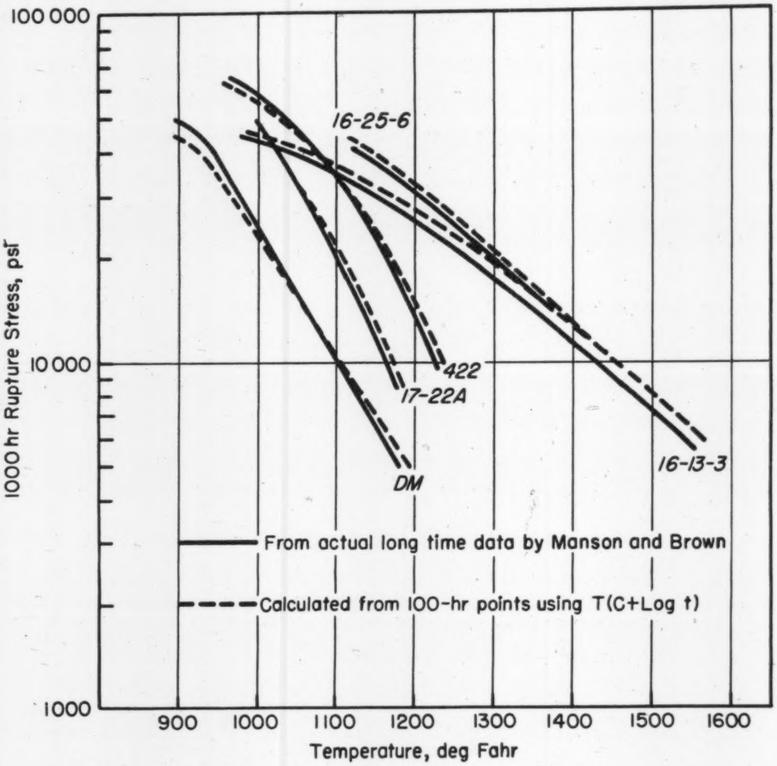

FIG. 10.—1000-hr Rupture Strength *versus* Temperature for Alloys Tested. Comparison of Curves Obtained from Long Time Data and Curves Predicted by $T\ (C + \log t)$.

The parameter $T(C + \log t)$, where T is the absolute temperature and t the rupture time in hours, is based on the rate process theory and is relatively simple since the value of C is for practical purposes equal to 20 for the vast majority of alloys, from aluminum base to iron, nickel, and cobalt base. The outstanding exception is the 12 per cent chromium-iron base alloys where $C = 25$ to 30.

In defense of this latter parameter, the accompanying Fig. 10 has been prepared showing the 1000-hr rupture strength of the alloys studied in this paper plotted against temperature. The solid lines represent the actual 1000-hr strength obtained from the authors' long time data, while the dotted lines have been calculated using only the 100-hr strength and the parameter $T(C + \log t)$.

This was done in the following manner. Master curves were constructed by plotting the 100-hr strength against $T(C + \log t)$ where $C = 20$ for all the alloys except the 12 per cent Cr alloy 422 for which previous data had shown $C = 30$. From the master curves the 1000-hr strengths were then read off at the appropriate values of the parameter. It is observed that the 1000-hr strength has thus been predicted for several temperatures with an accuracy about equivalent to experimental error from only a few 100-hr points. To obtain this data in the conventional manner would require several tests at each of several temperatures extending out to at least a few hundred hours.

While the authors' linear temperature parameter does a good job of correlating the data over the range of time available, it is based on the assumption that the constant stress lines converge to a single time at some low temperature usually in the vicinity of 700 F. This is contrary to experience since at low temperatures a slight change in stress causes a very large change in rupture time. The parameter $T(C + \log t)$ on the other hand is based on relations which have considerable basis in theory, so that in making extrapolations out to very long times where no data is available, such as 10 or 20 yr, one might have greater confidence in using this parameter.

It should be emphasized, however, that any method of obtaining long-time data from short time tests is an extrapolation and in consequence will be more accurate the longer the actual testing times are. Wide use has been made of the parameter $T(C + \log t)$ in various General Electric laboratories and elsewhere, and it has proven a valuable aid in obtaining the most information from available test data.

MESSRS. S. S. MANSON AND W. F. BROWN, JR. (*authors' closure*).—The authors wish to thank both Mr. Marble and Mr. Miller for their interesting discussion of the problem and for pointing out certain applications in which the authors' parameters appear to be particularly suitable.

The exact definition of this problem seems to be our major point of disagreement. Mr. Marble has suggested that certain features in the application of a parameter or in the testing equipment contribute toward its suitability. We feel, however, that there is only one logical absolute standard for judgment, namely, does the parameter correlate the available data for a given alloy within the range of scatter as determined by careful tests. We believe that the linear parameter most nearly satisfies this requirement.

Mr. Marble's first point is that certain creep equipment does not readily permit the direct determination of constant nominal stress curves. It should be pointed out that the major reason for determining these curves was to obtain a direct check on the assumptions involved in the formulation of the various time-temperature parameters. However, once confidence has been obtained in a given parameter these curves and their associated constants may be obtained indirectly from the conventional isothermal plots. Thus, a number of isothermal curves could be established, say between 30 and 300 hr, and a cross plot made of stress *versus* temperature with time as parameter. From this representation the constant nominal stress curves may be plotted. This is essentially the procedure used by Manson and Hafred in their analysis of the literature data.

Mr. Marble's second point is that fewer tests are required to establish a master curve based on the Larson and Miller parameter. This is true, but we wish to emphasize that a corresponding sacrifice in accuracy must be accepted. We feel that in most cases the sacrifice in accuracy is not justified by the few tests which are saved. In addition, no conclusive evidence has been presented which would serve as a guide in selecting the proper value of C in the parameter $T(C + \log t)$. In fact, it appears that as additional data are gathered on a wide variety of materials the number of values which C may assume increases steadily. If C must be determined directly, then the savings in tests required is greatly reduced.

Mr. Marble's third point is that the master curve based on the linear parameter is subject to variation if plotted by different individuals because of the extrapolation required in obtaining the two material constants. This is true; however, it should not be implied that this variation will appreciably affect the accuracy of correlation. The individual values of the material constants are not critical provided they occur in the right combination. This correct combination can readily be obtained by extrapolation of the lines of constant nominal stress. The selection of a point of intersection has not been found to be critical or to require great experience. Furthermore we should like to point out that the comparison between two heats (or between two materials) can easily be made using the linear parameter even though the master curves are different. Thus, it is only necessary to make the comparison on the basis of the conventional isothermal curves as developed from the master curves.

As is indicated by Mr. Marble, in most cases there is a variation in stress rupture properties from heat to heat. This is recognized by the authors but not as a reason for using a less accurate parameter. The variation from heat to heat varies with the alloy, some being much more uniform than others. If a variation or scatter band is to be accurately determined then we must use an accurate measuring device. The problem is a statistical one and requires a relatively large number of carefully conducted tests and an accurate parameter in order to reduce to a minimum all variations except those introduced by the primary variables of alloy melting and fabrication practice.

Mr. Miller has indicated that the parameter $T(C + \log t)$ has considerable basis in theory, being related to the rate process equation. While we do not deny that the parameter is in some way related to rate theory, the derivation given by Larson and Miller (1) seems to be quite arbitrary. Referring to the derivation as presented by Larson and Miller, the rate equation is stated as

$$r = Ae^{-Q/RT}$$

where r is a rate. The statement is then made that the time to rupture depends upon the summation of the creep rates to rupture and therefore the above equation can be rewritten in terms of rupture time, t, as follows

$$1/t = Ae^{-Q/RT}$$

First, it is not clear to us how the rupture time can be taken as a summation of creep rates nor is it clear how the rate process theory can be applied in any form without stating specifically a process for which a rate can be directly written.

Mr. Miller's statement that the value of C in the parameter $T(C + \log t)$ can in all cases be taken as 20 with the exception of the 12 per cent chromium base alloys is apparently in disagreement with facts presented in the authors' paper and in the paper published previously by Manson and Hafred. The authors' would like to call attention to the data for DM steel shown in their Fig. 1. It is evident that taking C-20 yields very poor correlation and for this low-alloy steel even the optimum value of C did not yield satisfactory results. Furthermore, Mr. Marble implied in his discussion that the value of C may vary from 15 to 30 depending on the material.

Referring to Mr. Miller's representation showing the dependence of the 1000-hr rupture strength on temperature it should be pointed out that this type of analysis represents the performance of the parameter under only one rather restricted set of conditions. Thus, a prediction of 1000-hr data from 100-hr results could probably be obtained satisfactorily by extrapolation of the isothermal curves on a conventional log log plot. A satisfactory parameter should permit extrapolation over a much wider range of the experimental variables. In order to test the parameter $T(C + \log t)$ under what we feel to be conditions more indicative of its suitability, we have used it to predict the 1000-hr data for DM steel from the 10-hr data. For this purpose C was taken as recommended by Mr. Miller to be equal to 20, and the predictions are made on the basis of a master curve constructed for the 10-hr rupture strength. In the accompanying Fig. 11 the calculated 1000-hr rupture strength is compared with the experimentally established curve. Errors in stress now amount to as much as 25 per cent depending on the temperature. In order to determine the corresponding errors in time, we have used the 10 hr rupture strength master curve based on

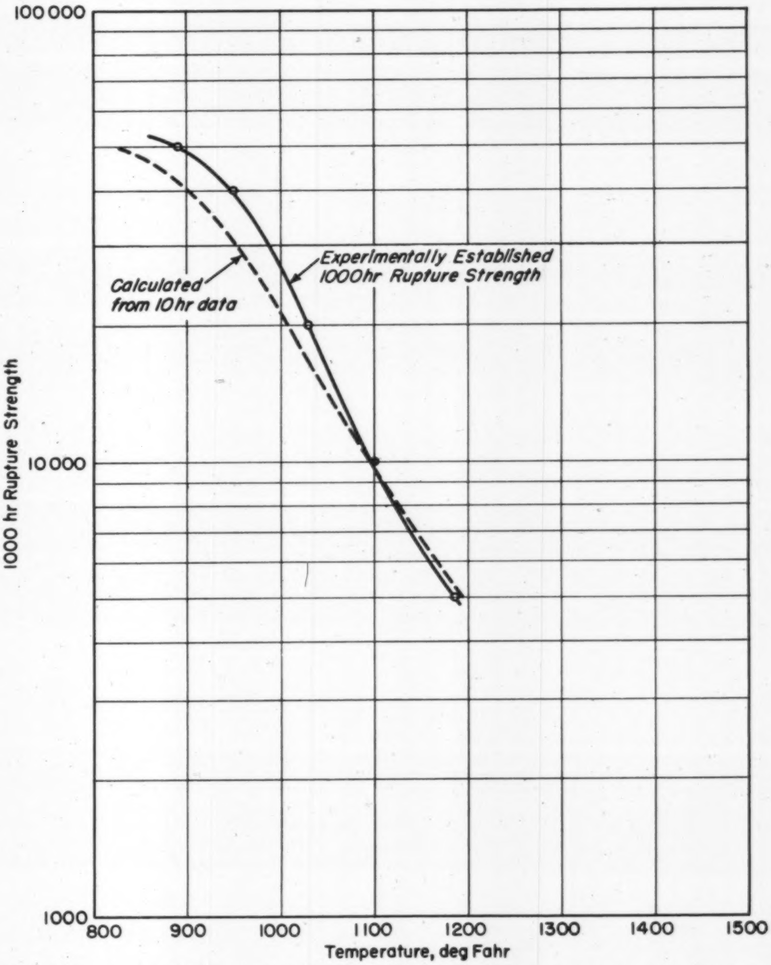

FIG. 11.—Experimentally Determined 1000-hr Rupture Strength Compared with Results Predicted from 10-hr Data Using the T $(20 + \log t)$ Parameter.

$T(20 + \log t)$ to calculate lines of constant nominal stress for the DM steel data shown in the authors' Fig. 1. Added to this representation (Fig. 12) are the results based on the 100 hr rupture strength master curve (that used by Mr. Miller). The calculated curves must necessarily agree with the experimental data in the region of 10 and 100 hr since the master curves were drawn specifically to fit these data.

The calculated curves generally show good agreement with the experimental data only at the lower stresses. At the higher stresses the agreement is poor. In this region the curves calculated from the 10-hr data agree best with the experimental points at short times while those calculated from the 100-hr data agree best with the experimental points at long times. This type of behavior might be expected from an examination of the

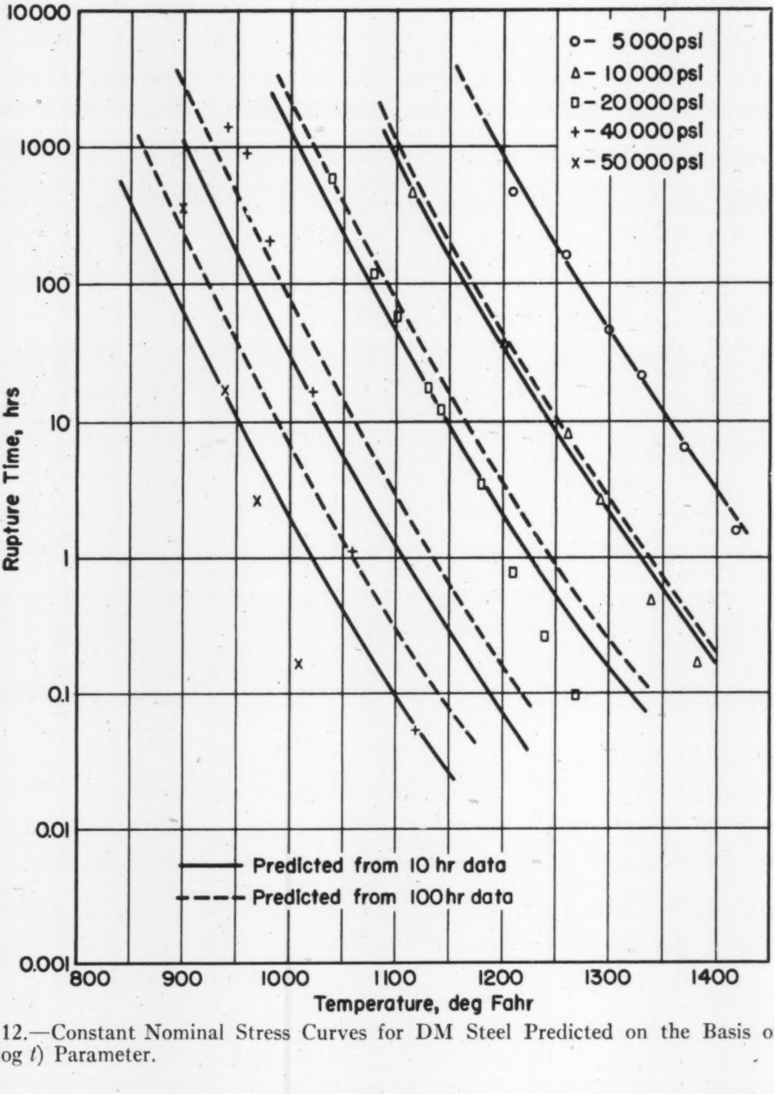

FIG. 12.—Constant Nominal Stress Curves for DM Steel Predicted on the Basis of the $T(20 + \log t)$ Parameter.

master curve for C = 20 presented in the authors' Fig. 6. It will be noted that the points are grouped closely at the low stresses indicating the parameter $T(20 + \log t)$ is correlating best in this region. At the high stresses, considerable spread exists and the points show a systematic deviation from the average curve. At these stresses the parameter $T(20 + \log t)$ cannot be used for accurate extrapolation over any appreciable time range.

Mr. Miller further states that the linear parameter is based on the assumption that the lines of constant nominal stress converge at some low temperature. We should like to refer to the analysis made in the authors' present paper which points out that the constant nominal stress data are faired by the best curve possible and that the tangents to these curves in the 30 to 300-hr range are used to establish the parameter constants. The authors do not claim that the constant nominal stress data are linear to times in the vicinity of $\log t_a$, this usually being a number greater than 8 (100,000,000 hr or approximately 11,000 years) but rather that is the practical time range the curves may be replaced by their tangents to a sufficient degree of accuracy. The authors have also presented a more general parameter which is able to take into account curvature of the constant nominal stress lines. However, it should be emphasized that for the materials examined thus far when curvature does occur it appears to be most pronounced at the extremely low times.

The authors wish to emphasize that they feel none of the parameters so far presented including their own can truly represent the basic behavior of a complicated alloy (such as a low-alloy steel) over extremely wide ranges of both time and temperature. It must be realized that the conception of a correlating parameter in the relatively simple forms thus far proposed is at best a tool which can permit the shortening of stress rupture testing times in ranges of practical application. The extrapolation of say 1000-hr data to yield 175,000 hr (20 yr) results might be an extremely dangerous procedure at the present state of knowledge, and is certainly not recommended. However, if a reasonable extrapolation is to be made it would seem logical to use the parameter which yields the best correlation of the available data.

THE MEASUREMENT OF DIRECTIONAL STRENGTH IN STRAIGHT- AND CROSS-ROLLED STRIP STEEL BY THE NAVY TEAR TEST*

By Helen D. Hoover[1]

Synopsis

This work comprised an examination of two lots each of straight- and cross-rolled commercial quality 1085 strip steel. Bwg No. 9 in thickness, and was part of an investigation of the suitability of these materials for commercial application. Its purpose was to measure the directional differences that are known to exist between the two rolling types but that had not been definitely determined because a testing method, adequate to measure them quantitatively, has been lacking.

The following methods were applied:

1. Chemical analysis, microscopic examination, and the ball test. These gave partial information.

2. Axial tension test, reverse bending fatigue test, cathodic etching, and a magnetometric study. These added nothing pertinent to the basic problem.

3. The Navy tear test. This was satisfactory and showed directionality and type of rolling in the hardened-and-tempered strip steel. It gave a quantitative strength distribution in numerical values around a hypothetical center point in the strip.

For the material used, the tear test showed:

(*a*) Directionality was most pronounced in the straight-rolled strip.

(*b*) Strength apparently increased with rolling effect.

(*c*) The over-all strength of the two types of strip, when directionality was nullified by averaging, was approximately the same.

A prevailing opinion is that cross-rolled steel, which has been equally reduced along its two major axes, is uniformly strong in any direction, and that straight-rolled steel is strongest when stressed in its rolling direction and weakest when stressed transversely to it. That differences exist between the two rolling types is accepted, but a testing method, adequate to measure them quantitatively, has been lacking. Claims have been made that various tests will show these variations but supporting data and literary references are rare.

This investigation was undertaken with the main object of measuring directional differences in straight- and cross-rolled strip steel by a method adequate to definitely show behavior differences on the basis of rolling type, other things being reasonably equal. A secondary objective was to check the value of various other tests in this application.

A comprehensive study of two lots each of commercial quality straight- and cross-rolled SAE 1085 strip steel, Bwg No. 9 in thickness, was made by the following methods: chemical analysis, microscopic examination, the ball test, the axial tension test, the reverse bending fatigue test, cathodic etching, magnetometric study, and the Navy tear test.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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TABLE I.—CHEMICAL COMPOSITION OF STEELS TESTED.

	Chemical Composition, per cent								
	C	Mn	P	S	Si	Cu	Ni	Cr	Mo
Straight-rolled	0.88–0.89	0.84–0.88	0.010–0.015	0.030–0.035	0.18–0.23	<0.10	<0.10	<0.10	<0.10
Cross-rolled	0.82–0.89	0.97–1.00	0.010–0.020	0.030–0.035	0.19–0.20	<0.05	<0.10	<0.10	<0.06

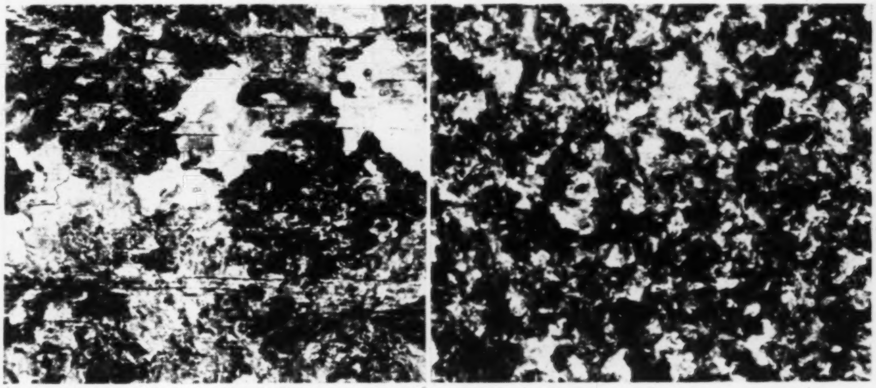
(*a*) (*b*)

FIG. 1.—Longitudinal Sections, 1085 Strip Steel as Rolled. Etchant, 2 per cent Nital (× 100)

(*a*) Straight-rolled strip, showing irregular grain size and pearlitic structure.
(*b*) Cross-rolled strip, showing regular grain size and pearlitic structure.

MATERIAL

Description of Steel:

Four strips of Bwg No. 9 commercial quality SAE 1085 strip steel, two each of straight- and cross-rolled, were tested. Extension from ingot to strip was approximately 210 × for the straight-rolled; for the cross-rolled, extension from ingot to slab was approximately 13 × and, at 90 deg to the primary rolling direction, extension from slab to strip was approximately 19 ×.

Quality:

Inspection revealed no surface defects, laminations, or other detrimental irregularities.

Gage:

Thickness was 0.146 to 0.153 in. for the straight-rolled and 0.145 to 0.154 in. for the cross-rolled and was in the upper portion of the 0.148 ± 0.008 in. limits of Bwg No. 9. Edge-to-center variance was slight in the cross-rolled and not apparent in the straight-rolled.

Hardness:

As-rolled Rockwell hardness of the straight-rolled strip was C 17 to C 19; of the cross-rolled, C 27 to C 34.

Chemical Composition:

No differences in chemical composition greater than the permissible variations of the American Iron & Steel Inst. were found between edge and center locations or between the two strips of each rolling type. Table I shows the range of results obtained from eight samples from each type of strip.

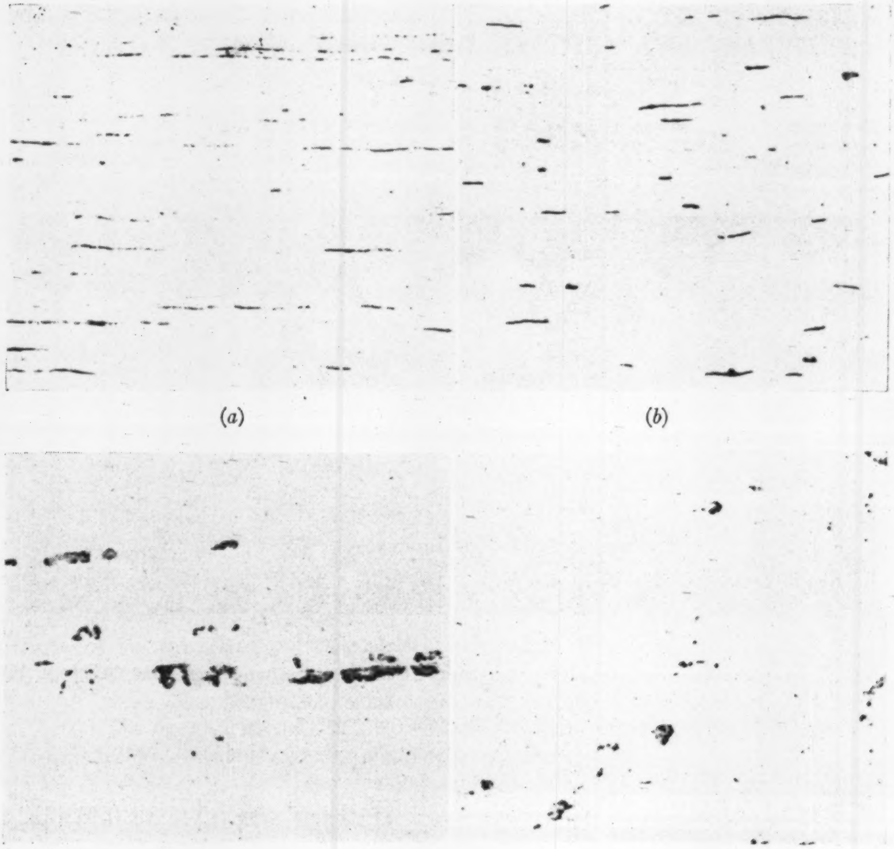
(a) (b) (c) (d)

FIG. 2.—Nonmetallic Inclusions in 1085 Strip Steel. Unetched (× 100).

(a) Distribution of nonmetallics in longitudinal cross-section of straight-rolled strip.
(b) Distribution of nonmetallics in cross-section parallel to final rolling direction of cross-rolled strip.
(c) Ribbonlike shape of inclusions in straight-rolled strip on plane parallel to surface.
(d) Irregular flat shape of inclusions in cross-rolled strip on plane parallel to surface.

Microscopic Examination:

Microstructure of both types of steel was pearlitic, with evidence of higher finishing temperature for the cross-rolled. Figures 1(*a*) and (*b*) show typical longitudinal sections of straight- and cross-rolled, respectively, the former exhibiting coarser pearlite, and larger and less even grains. Some intragranular cold work was observed in the straight-rolled strip at higher magnifications. At 250 × this was evidenced by a "rippling" parallel to the rolling direction, which passed freely from one colony of pearlite to the next, but not from grain to grain. At 1000 ×, these areas showed fracture and dislocation of the lamillae. Transverse sections of both types were similar to Fig. 1(*b*), the straight-rolled being coarser. Present background does not permit attempts to relate these slight differences with directionality in the heat-treated condition, as shown by these tests.

Surface decarburization was partial and not severe enough to reduce exterior Rockwell C hardness materially below interior hardness after heat treating. Mill-edge carbon loss was not excessive.

Nonmetallic inclusion orientation and frequency differed with the types of rolling; segregation, elongation, and number of inclusions were greater in the straight-rolled strip examined. Inclusion rating was made by Method A of the Standard Recommended Practice for Determining the Inclusion Content of Steel E 45).[2] Eight longitudinal specimens, 1 in. by full strip thickness, were taken from each of the four strips at even increments from mill edge to mill edge, and their whole area surveyed. All fields were of the thin type with the exception of two which were among the better fields and thus were not recorded. Average results were: for the straight-rolled, A 5.0, B 0.1, C 0.2, D 2.0; for the cross-rolled, A 3.0, B 0.4, C 0.3, D 1.5. Figures 2(*a*) and (*b*) show the types observed in longitudinal cross-sections. Figures 2(*c*) and (*d*) were taken on sections parallel to and approximately 0.050 in. beneath the strip surface. Transverse cross-sections showed the expected "peppered" effect with some short stringers in the cross-rolled. Very clean to very dirty fields were observed in both types of strip, the cross-rolled showing a more homogeneous distribution of the nonmetallics than the straight-rolled. Heaviest concentrations were not confined to central sections, and sporadic segregations from surface to surface were noted.

The long, discontinuous, ribbonlike stringers, which were typical of the straight-rolled, seem more likely to contribute to directional weakness than the shorter inclusions, pancaked in flat section, which are typical of the cross-rolled. How strong the effect of the inclusions may be in relation to other factors which influence directional behavior was not revealed by this study.

To summarize, the material was of satisfactory quality and reasonable uniformity. The only notable differences between the straight- and cross-rolled lots were in as-rolled hardness, manganese content, and nonmetallic frequency and orientation.

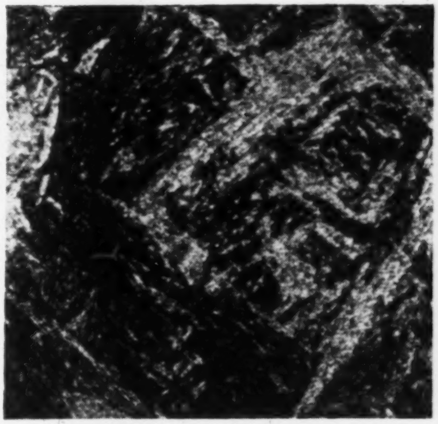

FIG. 3.—Tempered Martensitic Structure of Hardened-and-Tempered Specimens for Mechanical Tests. Etchant, 4 per cent Picral (× 1000).

HEAT TREATMENT

It was thought that a tempered martensitic structure of about Rockwell C 42 would give suitable physical properties and also reveal any sensitivity of the material to nonmetallic inclusion differences. All mechanical test specimens were heat treated as follows:

1. Austenitize in neutral salt, 1575 F, 10 min total time.
2. Quench in neutral salt, 350 F, 2 min.
3. Air cool to room temperature.
4. Wash with hot alkali cleanser; dry.
5. Furnace-temper, 820 F, 1 hr.

The Rockwell hardness obtained was C 41 to C 45. Figure 3 shows a typical heat-treated microstructure.

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[2] 1952 Book of ASTM Standards, Part 1, p. 1477.

PRELIMINARY TESTS

Ball Test:

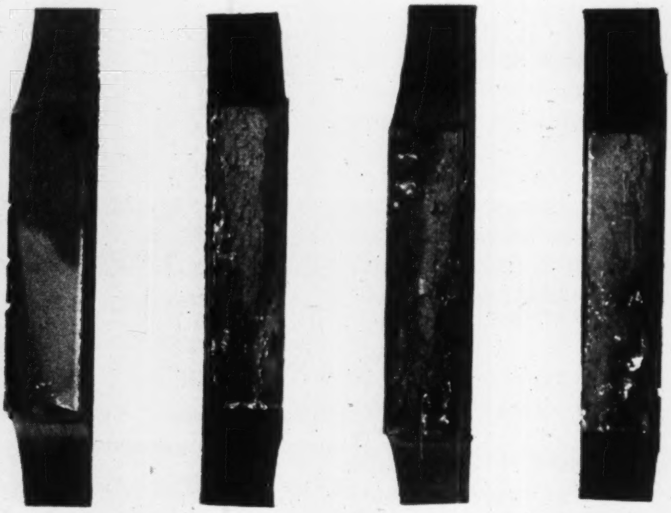
The ball test is made by pressing a 1-in. hardened steel ball into a smoothly reamed and chamfered 0.75-in. hole in a section of strip supported during the test by a hardened ring of 3-in. inside diameter on which the hole in the sample is centered.

The straight-rolled strip fractured straight along the rolling lines; the cross-rolled produced either random or curved breaks, unrelated to the rolling. Although this test differentiated between the two types of strip by fracture, no quantitative interpretation of the maximum loads to produce fracture was attempted because of the numerous variables and the small amount of present knowledge on which to base such an attempt.

TABLE II.—AVERAGE TENSILE PROPERTIES.[a]

Property	Straight-Rolled			Cross-Rolled		
	Parallel	Transverse	Average	Parallel	Transverse	Average
Tensile strength, psi	201 500	203 500	202 500	206 600	206 100	206 300
Yield strength, psi	164 100	165 400	164 800	167 700	175 300	171 500
Ratio of yield strength to tensile strength	0.81	0.81	0.81	0.81	0.85	0.83
Elongation in 2 in., per cent	9.0	7.5	8.0	7.0	6.5	7.0

[a] $n = 8$.



Parallel Transverse
Straight-Rolled

Parallel Transverse
Cross-Rolled

FIG. 4.—Reverse Bending Fatigue Test Fractures, Hardened-and-Tempered 1085 Strip Steel (× 2).

Axial Tension Test:

Axial tension specimens were cut parallel and transverse to the (final) rolling direction of the strip. Average results are listed in Table II. Statistical analysis of the data from which Table II was compiled showed no significant difference in tensile properties between (1) the straight- and cross-rolled steel, (2) the two lots of each type, (3) the edge and center sections of each strip, and (4) the parallel and transverse specimens. Fracture surfaces showed variations much like those of Fig. 4.

Reverse Bending Fatigue Test:

Polished specimens for reverse bending fatigue tests were similar in strip location to the axial tension specimens. These tests produced distinct fracture differences by location and rolling type, as shown in Fig. 4. Figure 5, which summarizes all fatigue test data, shows that the results obtained were contradictory.

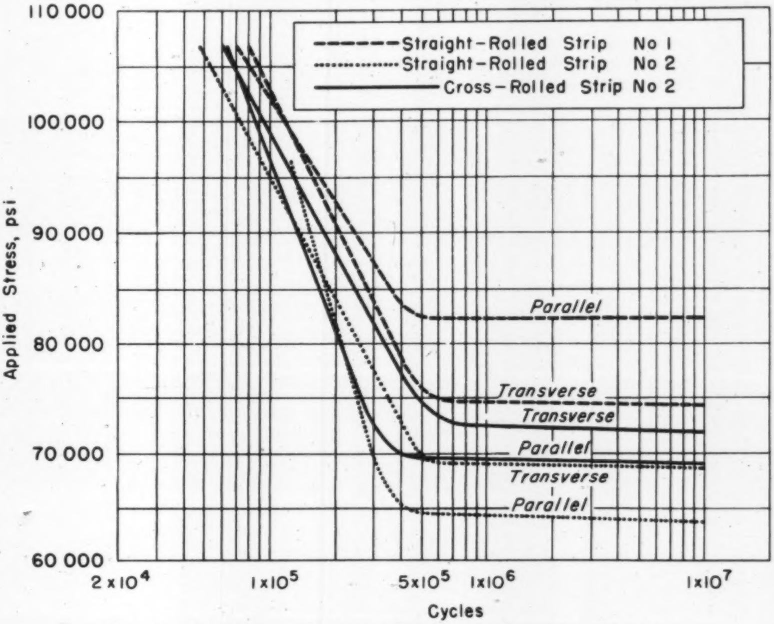


FIG. 5.—Summary of Reverse Bending Fatigue Test Results.

Magnetometric Study:[3]

Tests with a spring torque magnetometer did not separate the straight- and cross-rolled lots.

Cathodic Etching:[3]

Specimens prepared by cathodic etching revealed nothing pertinent to the problem that was not shown by chemical etching techniques.

NAVY TEAR TEST

Procedure:

The Navy tear test was developed by Kahn and Imbembo as a means of evaluating transition from shear to cleavage fracture in ship plate (**1,2,3,4**).[4] The specimen is shown in Fig. 6, with arrows marked *s* indicating loading direction.

Tests were run on a 60,000-lb capacity testing machine, with PD-1M deflectometer and automatic stress-strain recorder. Specimens were positioned as shown in Fig. 7. Static tensile loading at the rate of 0.10 in. per min was applied asymetrically, with initial eccentricity of 0.039 in., to fracture.

Thirty-six blanks were sawed from each strip to be tested. The diagrams of Fig. 8 show the sampling plan. Twice as many specimens were taken parallel to the rolling (0 deg) as transverse to it (90 deg), so that edge-to-center variance might be checked by varying the notch position. Angled specimens covered both directions from the rolling axes.

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[3] This should not be construed as criticism of these methods nor as a statement of limitation, since it is possible that both might contribute more if the investigation were carried further.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 736.

The importance of accurate directional sampling for this application of the Navy tear test cannot be overemphasized.

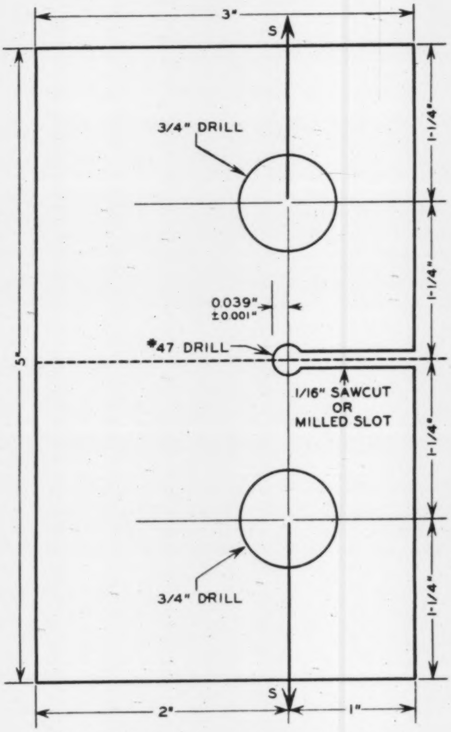

FIG. 6.—Navy Tear Test Specimen.

The edges to be notched were ground. A jig-bored fixture was used to locate accurately the 0.75-in. support holes and 0.078-in. drill hole in relation to each other and to the ground edge. Notches were milled in groups of six. No further machining was required after heat treatment.

Maximum load in pounds was recorded from the dial of the testing machine, and work absorbed to fracture in foot-pounds was computed from the area under the curve. Because variations in thickness and fracture length were small, calculations to standard area or thickness are not reported. Extension in inches was read from the curve.

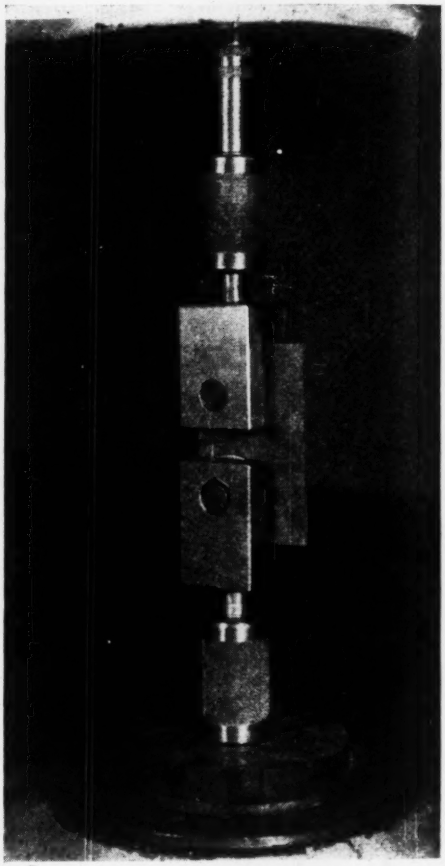

FIG. 7.—Navy Tear Test Specimen and Fixtures in Position for Testing.

Spread was not excessive. Data were averaged by location in the strip for each angular position and lot of material. No significant differences were shown between locations or lots of each rolling type. Individual data and detailed breakdowns have been omitted as

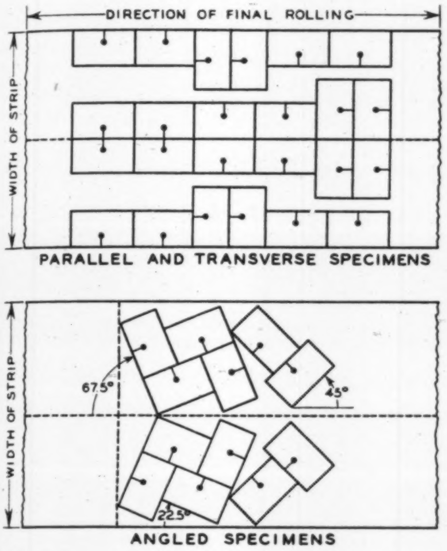

FIG. 8.—Plan of Sampling for Navy Tear Test.

unnecessary. "Work absorbed to fracture" refers to initial fracture.[5]

Table III shows average property values, individual maxima and minima, and percentage differences for all parallel and transverse specimens. The spread for these positions is representative of that for other angular positions tested. It is interesting that, although percentage differences between parallel and transverse specimens of each type are marked, the average of the two positions shows the over-all strength of the straight- and cross-rolled to be almost the same.

Table IV shows averages of all tear tests. The 0 and 90 deg values are the same as in Table III. The over-all averages are not importantly different when the fracture variables related to the angular positions are considered. (See section on Fracture Indications.)

Discussion of Data:

Strength Distribution.—Some of the data from Table IV have been expanded to give the strength-distribution curves of Fig. 9. (The effect of extension is reflected in the work absorption values so it was not graphed separately.) The position of the specimens in relation to

TABLE III.—COMPARISON OF AVERAGED TEAR PROPERTY VALUES, PARALLEL AND TRANSVERSE SPECIMENS.[a]

Tear Property	Straight-Rolled			Cross-Rolled		
	Parallel	Transverse	Average	Parallel	Transverse	Average
Maximum load, lb	20 150	14 450	17 300	18 100	16 900	17 500
Difference[b]	+39.5	...	...	+7.1	...	...
Difference[c]	...	...	...	...	...	+1.2
Minimum	19 250	13 650	...	16 100	15 650	...
Maximum	20 950	15 850	...	18 900	17 800	...
Work absorbed to fracture, ft-lb	98	45	71.5	75	64.5	70.0
Difference[b]	+117.8	...	...	+16.3	...	...
Difference[c]	...	...	+2.1	...	...	...
Minimum	78	41	...	63	53	...
Maximum	104	52	...	82	74	...
Extension, in	0.1110	0.0755	0.0935	0.0970	0.0905	0.0940
Difference[b]	+47.0	...	...	+7.2	...	...
Difference[c]	...	...	...	...	...	+0.5
Minimum	0.0975	0.0700	...	0.0825	0.0800	...
Maximum	0.1200	0.0850	...	0.1050	0.1000	...

[a] $n = 32$, 0 deg; $n = 16$, 90 deg.
[b] Percentage difference between parallel and transverse.
[c] Percentage difference between straight-rolled and cross-rolled averages.

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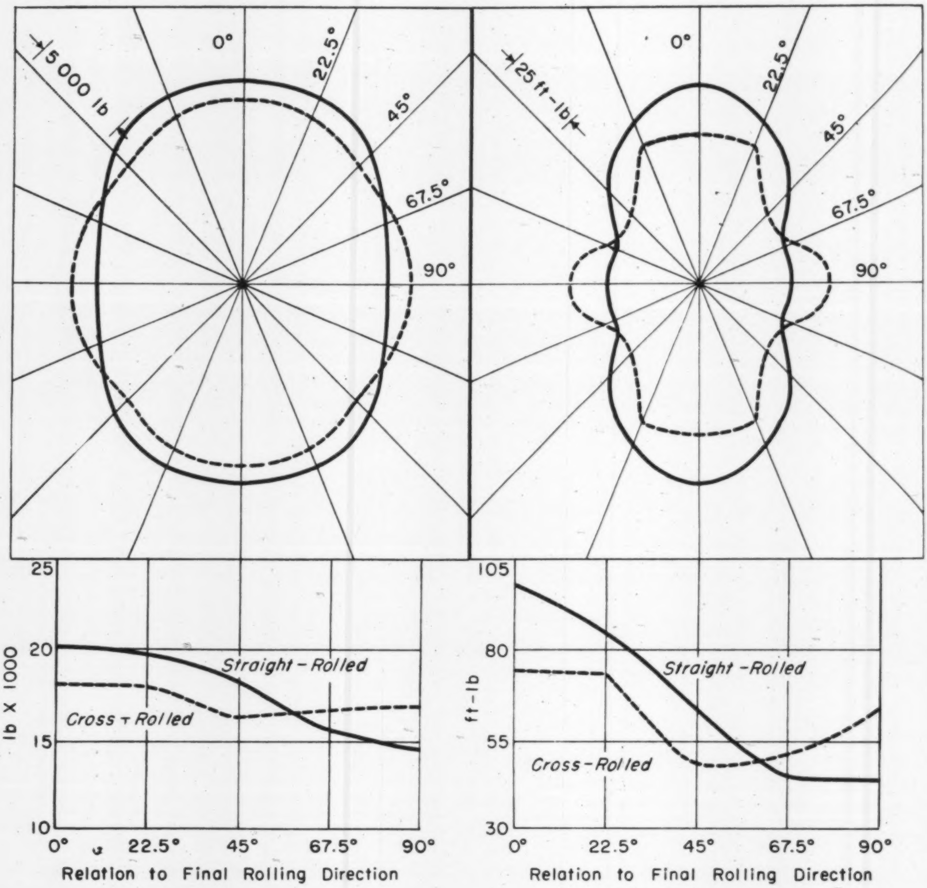
[5] No measurable tearing occurred during the fracture of any specimen, so energy to propagate fracture is so small a part of the whole energy that it may be considered negligible. Stress analysis of the specimen, discussed later in this paper, showed that the area involved in initiating the fracture was very small and that the thickness at the base of the notch was the important dimension in these tests. In subsequent work, values will be presented on the basis of standard thickness of 1 in.

TABLE IV.—AVERAGED TEAR PROPERTY VALUES, PARALLEL, TRANSVERSE, AND ANGLED SPECIMENS.[a]

Tear Property	Angle of Specimen, Relation to Final Rolling Direction, deg					Over-all Average
	0	22.5	45	67.5	90	
STRAIGHT-ROLLED						
Maximum load, lb	20 150	19 800	18 300	15 450	14 450	17 650
Work absorbed to fracture, ft-lb	98.0	85.5	64.0	45.0	45.0	67.5
Extension, in.	0.1110	0.1025	0.0825	0.0695	0.0755	0.0880
CROSS-ROLLED						
Maximum load, lb	18 100	17 900	16 300	16 700	16 900	17 200
Work absorbed to fracture, ft-lb	75.0	74.0	48.0	51.5	64.5	62.0
Extension, in.	0.0970	0.0870	0.0705	0.0735	0.0905	0.0835

[a] $n = 32$, 0 deg; $n = 16$, 90 deg; $n = 8$, all other positions.



Maximum Load, lb

Work Absorbed to Fracture, ft-lb

FIG. 9.—Strength-Distribution Curves, Hardened-and-Tempered 1085 Strip Steel, Straight- and Cross-Rolled.

the (final) rolling direction is indicated by angular positions, where 0 deg is parallel to the rolling and 90 deg is transverse to it. The polar graphs show a 360-deg distribution of strength around a hypothetical center point in the strip; the rectilinear curves cover 90 deg only.

Some conclusions that may be drawn from Table V follow:

1. The maximum strength of the straight-rolled material was parallel to the rolling direction and the minimum transverse to it. The high maximum (I) of the cross-rolled was parallel to the final rolling direction, the lower maximum (II) was parallel to the primary rolling direction, and the minimum was at 45 deg to the rolling axes, where the rolling effect should be very slight if the primary and final extensions were approximately the same.

Therefore, strength seems to increase with rolling effect.

2. The straight-rolled material was stronger than the cross-rolled through the two thirds of the strip section (0 to 60 deg) extending from the rolling direction; the cross-rolled was stronger through the one third (60 to 90 deg) extending from the direction at right angles to the final rolling.

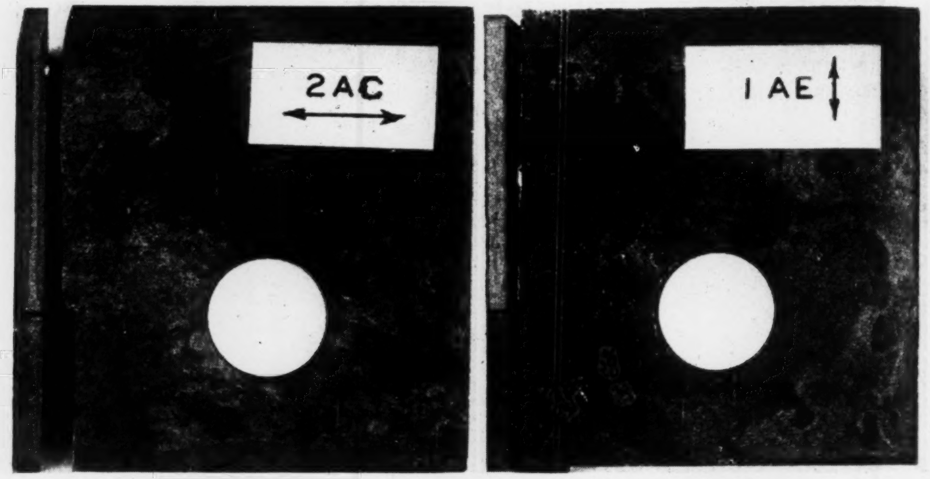
Therefore, the cross-rolled strip had a more even strength distribution than the straight-rolled, although it was weaker throughout two thirds of the strip section.

3. Parallel to the rolling direction, the straight-rolled strip was 40 per cent higher in maximum load and 118 per cent higher in work absorbed to fracture than transverse to it, whereas the cross-rolled was, respectively, only 7 and 16 per cent higher. At maximum strength the straight-rolled strip was 40 per cent higher in maximum load and 118 per cent higher in work absorbed to fracture than at minimum, whereas the cross-rolled was, respectively, only 11 and 57 per cent higher. The maximum values for the straight-rolled strip were 11 per cent higher in maximum load and 29 per cent higher in work absorbed to fracture, with the corresponding straight-rolled minimum values being 11 and 6

TABLE V.—SUMMARY OF TEAR STRENGTH DISTRIBUTION (FROM TABLES III AND IV AND FIG. 9)

	Maximum Load		Work Absorbed to Fracture	
	Position	Pounds	Position	Foot-Pounds
Straight-rolled				
Maximum	0 deg	20 150	0 deg	97.0
Minimum	90 deg	14 450	67.5 to 90 deg	45.0
Average	...	17 650	...	67.5
Comment	Stronger through ⅔ of sheet next to 0 deg axis			
Ratios	Parallel to transverse	1.40	Parallel to transverse	2.18
	Maximum to minimum	1.40	Maximum to minimum	2.18
Cross-rolled				
Maximum I	0 deg	18 100	0 deg	75.0
Maximum II	90 deg	16 900	90 deg	64.5
Minimum	45 deg	16 300	45 deg	48.0
Average	...	17 200	...	62.0
Comment	Stronger through ⅓ of sheet next to 90 deg axis			
Ratios	Parallel to transverse	1.07	Parallel to transverse	1.16
	Maximum I to minimum	1.11	Maximum I to minimum	1.57
Straight- and cross-rolled				
Equal	60 deg	16 500	60 deg	50.0
Ratios of straight-rolled to cross-rolled				
Maximum	1.11		1.29	
Minimum	0.89		0.94	
Average	1.03		1.09	

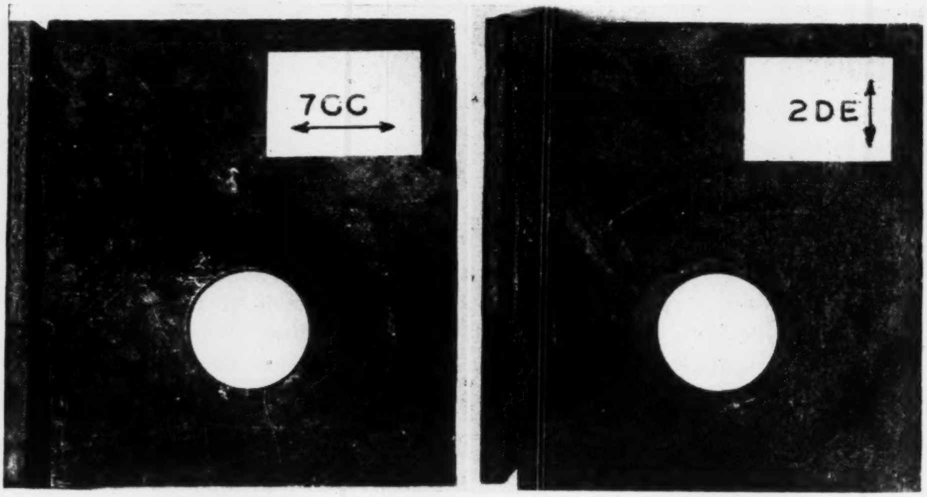
per cent lower than the respective cross-rolled minimum values.



Parallel

Transverse

Straight-Rolled



Parallel

Transverse

Cross-Rolled

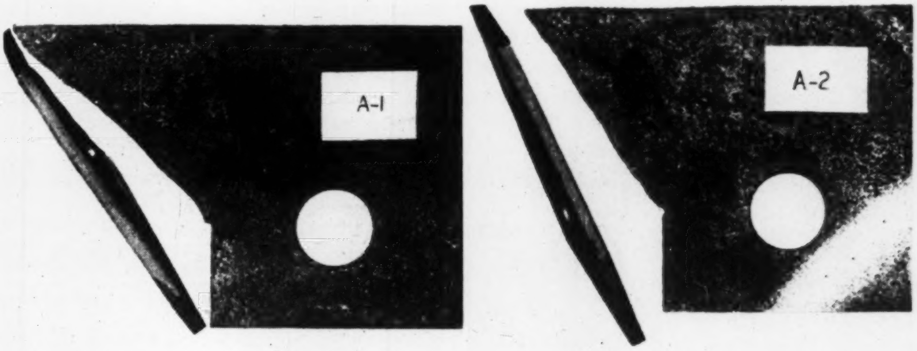
FIG. 10.—Typical Navy Tear Test Fractures, Parallel and Transverse Specimens, Straight- and Cross-Rolled Strip.

Therefore, the straight-rolled strip was stronger than the cross-rolled at maximum and weaker at minimum, had wider range, and showed greater strength advantage at maximum than disadvantage at minimum.

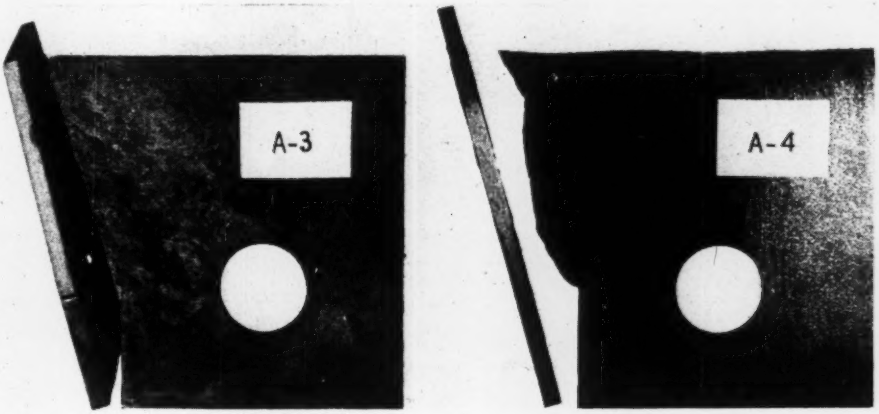
4. When tear property values from the five angular positions tested were averaged, the straight-rolled was only 3 per cent higher in maximum load and 9 per cent higher in work absorbed to fracture than the cross-rolled. Analysis by statistical methods indicates that this could have occurred by chance alone.

Therefore, the over-all strength of the

two types of strip, when directionality was nullified by averaging, was approximately the same.



(*a*) 22.5 deg from parallel. (*b*) 45 deg from parallel.



(*c*) 67.5 deg from parallel. (*d*) Irregular break.

FIG. 11.—Typical Navy Tear Test Fractures, Angled Specimens, Straight-Rolled Strip.

In the types of strip steel examined, and presumably in other types, sufficient specimens taken at 22.5 deg intervals in five positions starting parallel to the final rolling or rolling direction (0 deg) and tested as described here will give a quantitative strength distribution in terms of "maximum load, lb," around a hypothetical center point, with no calculations and no further work except plotting maximum load against angular position, provided thickness and fracture length do not vary excessively. Maximum differences of about 5 per cent were present in the specimens tested here, and did not significantly affect the results.

Fracture Indications.—In most cases, the autographic curves showed little or no indication of plastic flow before maximum load was reached, where the break was initiated and then propagated to complete failure "instantaneously."

The curve showed no evidence of tearing after maximum load was reached. Although these fractures all were completed abruptly, they were not of a wholly brittle nature. Shearing was present in some straight-rolled and all cross-rolled fractures, and it is a reasonable assumption that some small amount of tearing may have occurred. This examination was planned to determine directionality in relation to rolling type only, so no attempt was made to evaluate per cent of shear, notch sensitivity, or any small amount of energy to propagate fracture.

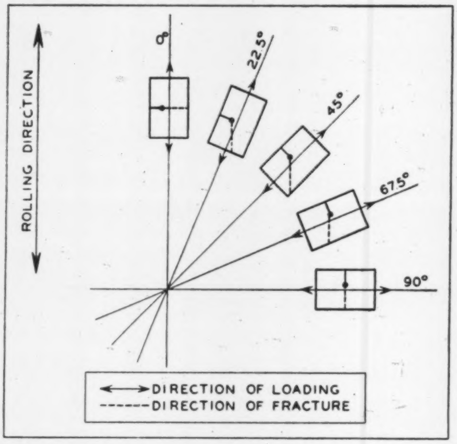


FIG. 12.—Direction of Loading and Fracture in Navy Tear Test, Straight-Rolled Specimens.

Figure 10 shows typical fracture surfaces and edges for parallel and transverse specimens of both types of steel. Figure 11 shows surface, edge, and direction of fracture for angled straight-rolled specimens. The arrows in Fig. 10 indicate the (final) rolling direction. Letters and numerals are specimen numbers not considered in this discussion. In Fig. 11, angular position of A-4 is omitted, this irregular type of break having occurred in two specimens cut at 45 and 67.5 deg. No pictures were taken of the angled cross-rolled specimens, which broke very similarly to the transverse cross-rolled.

From Figs. 10 and 11 it may be seen that:

1. Of the cross-rolled specimens, those cut parallel to the final rolling direction alone could be distinguished by position and only by careful examination of the fracture edge.

2. Straight-rolled parallel and transverse specimens and straight-rolled angled specimens could be readily separated in most cases by fracture surface and edge of the former, and by fracture direction of the latter.

3. Separation of straight- and cross-rolled material by this test is doubtful, unless complete sets of specimens are tested.

Numerous splits in the fracture surfaces were observed, which microscopic examination revealed were caused by nonmetallics, in some cases by discontinuous chains of minute inclusions. Since the test seems to be sensitive to very small defects, any serious flaws should be plainly revealed, if the sample tested should contain such a defect at or very near the line of fracture.

Figures 11 and 12 show that all straight-rolled specimens broke along or near the rolling lines except those cut and loaded parallel to the rolling direction (0 deg), where such a break normally could not occur. This directional fracture of the straight-rolled specimens is striking evidence of the strong directionality of this lot of material. No such fractures occurred in the cross-rolled and no exceptions were observed in the straight-rolled. (The two irregular straight-rolled fractures followed the rolling lines throughout part of the fracture length.) The difficulties of fracture proceeding at 22.5 deg to the loading direction are considerable, because such a break takes a path at 67.5 deg to the direction of maximum mechanical stress, which is at right angles to the loading direction. Such difficulties are less in specimens cut at 45 deg and still less in those cut at 67.5 deg. The fracture, which is apparently initiated in a very small area at right angles to loading, tends to follow the easiest way to complete failure. In these straight-rolled specimens, the differential between the weakest direction (with the rolling) and other directions in the strip is apparently large enough to overcome the mechanical obstacles to the formation of angular fracture under the test loading setup.

Stress Analysis of Specimen.—Figure 13 shows the photoelastic fringe pattern developed at the base of the notch on a catalin facsimile of the Navy tear test specimen after 60 sec under a load of 62 lb. The dots along the center line to the right of the base of the notch correspond to the positions plotted in the left portion of Fig. 14, which is a graphic representation of the two principal stresses in this location.

From Fig. 13 it may be seen that the stress is highest in a small angular range at the base of the notch. Figure 14 shows that it is very high on the center line at the notch base and decreases rapidly away from the notch along the center line to a relatively stable value. However, the difference between the principal stresses, as shown by the fringe value, decreases from a maximum at the notch to zero at approximately 0.099 in. and remains very small for some distance beyond. In this case, the low fringe value indicates almost complete biaxiality of the stress, as is shown by the approximate equality of the parallel and perpendicular stresses in Fig. 14.

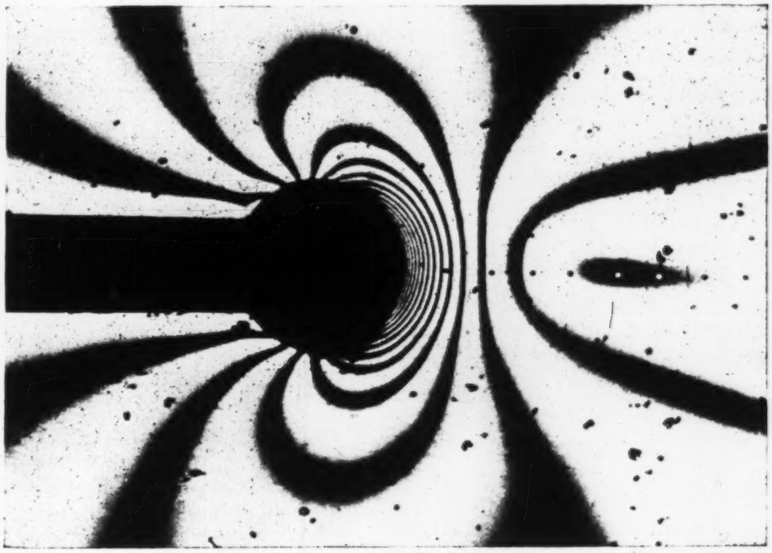


FIG. 13.—Photoelastic Fringe Pattern at Base of Navy Tear Test Specimen Notch (× 25 approximately).

One might expect some yielding to occur in a small section at the center of the base of the notch and then be inhibited by the biaxiality. The load would then rise to a maximum and the fracture take place abruptly. This concurs with the behavior of the specimens tear-tested, the autographic curves, and the appearance of the fractures.

At the base of the notch there is only

one stress, and the fracture must start inward at right angles to the loading direction. In the biaxial region, where there is no very dominant stress direction, the fracture might be expected to proceed in the weakest direction.

In the straight-rolled material, there is a considerable difference in strength in terms of maximum load between the minimum, that at 90 deg to the rolling direction where fracture parallels the rolling, and that at other locations in the strip. Fracture followed the rolling direction in this material in all cases where such a break was possible.

In the cross-rolled material, the load minimum at 45 deg to the rolling axes is only slightly lower than the values found for most other locations. Fractures proceeded directly across the specimen in a direction at right angles to the loading.

It seems that the combined effects of the strength differences in the two types of material and the approximate biaxiality in the tear test specimen along the center line in the region a short distance behind the base of the notch offer some explanation of the difference in fracture direction in the strip steel tested.

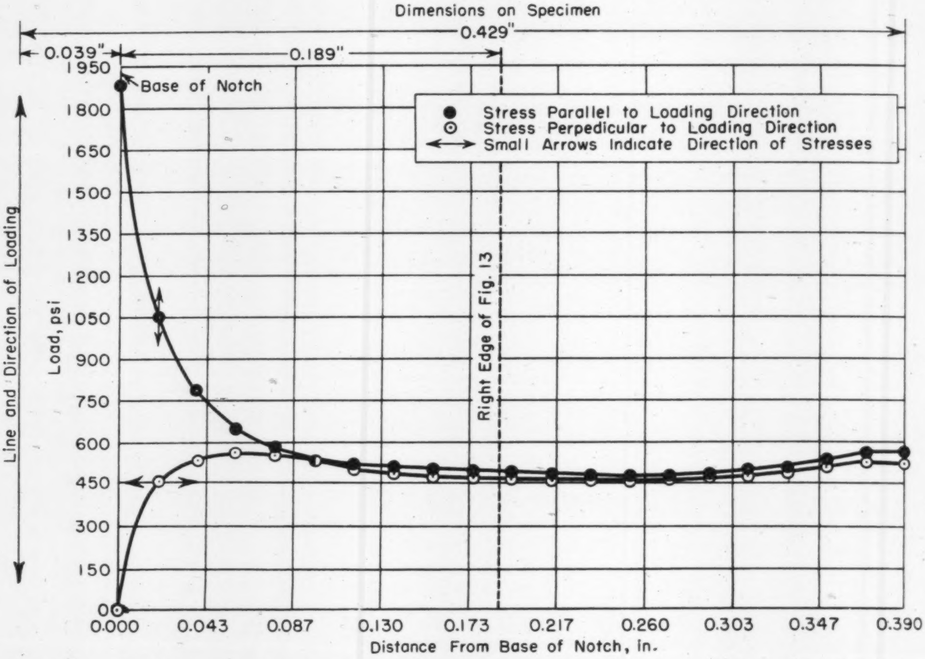


FIG. 14.—Graph of Two Principal Stresses at Base of Notch Along Center Line at Right Angles to Loading Direction.

Comment on Test Application.—This application of the Navy tear test is inexpensive, easy to perform, and may be simply interpreted in terms of directionality and relative strength distribution. Specimens are easily and quickly prepared. Observation indicates that slight warpage, surface scale, or decarburization does not invalidate results. No difficulty was experienced with specimens where the slot was slightly offset from the center of the notch drill hole.

The fixtures are simple. Centering on

the support pins by means of the spacing washers need only be approximate. The loading is as easy as in axial tension testing, and the adjustment prior to loading is easier, since the difficulties of securing proper alignment and firm gripping, encountered in tension testing of flat specimens, are absent.

Results from specimens of similar angular position and adjacent locations checked closely. Spread was slightly wider when specimens were located some distance apart in the strip. Three specimens exhibited eccentric behavior, for which no cause was found. Work absorbed to fracture showed greater scatter than maximum load, this being related to changes in extension for which the cause is not apparent. The results indicated an acceptable test accuracy which, however, was not computed because of the limited material for sampling.

SUMMARY

This application of the Navy tear test showed directionality and type of rolling in the 1085 hardened-and-tempered strip steel examined. It also gave a quantitative strength distribution in numerical values around a hypothetical center point in the strip.

Directionality in the straight-rolled strip was so marked that specimens loaded in the direction of sampling broke strikingly along the rolling lines, with the exception of those cut parallel to the rolling direction (0 deg), where such a break is normally impossible. The cross-rolled specimens broke at right angles to the loading regardless of the direction of sampling. Directionality in the straight-rolled material was shown both by fracture appearance and direction and by the strength-distribution curve. In the cross-rolled, it was shown by the curve only and was much less marked.

Strength appeared to increase with rolling effect. The highest and lowest strength values were exhibited by the straight-rolled strip, which was stronger throughout the section from 0 to 60 deg and weaker from 60 to 90 deg. The overall strength of the two types, when directionality was nullified by averaging, was about the same.

There are some indications that the Navy tear test, when applied to steel similar to that tested here, and possibly to other types, may give strength and rolling information both when the rolling axes are known and when they are not, within reasonable limitations. The ease and simplicity of applying the test, coupled with the large amount of information produced, point to possible great usefulness in the mechanical testing field.

Further work on similar material utilizing the Navy tear test, now in progress or planned at International Harvester Manufacturing Research, comprises the testing of curved sections, a study of the relation between mill practice and directionality (in cooperation with The Steel Company of Canada), and the effect of variations in heat treatment and chemistry. An attempt is also being made to use the test as a measure of drawability in deep-drawing quality sheet steel.

Chemical analysis, microscopic examination, and the ball test produced partial information, while the axial tension test, reverse bending fatigue test, cathodic etching, and a magnetometric study added nothing pertinent to the basic problem.

Acknowledgment:

The author wishes to thank the International Harvester Co. for granting permission to publish this paper, and to express appreciation to the Ingersoll Products Div. of Borg-Warner Corp. for supplying the steel. Appreciation is due to R. E. McGee, A. S. Jameson, J. H. Clark, J. A. Halgren, and R. H. Pinkel, of the Supervisory Staff of International Harvester Manufacturing Research, for their encouragement and criticism. The author is particularly grateful to Mr. Jameson, who first suggested the Navy tear test as a possible method for use in this study. She also wishes to thank her coworkers in the laboratories, whose fine cooperation, painstaking efforts, and helpful suggestions were largely responsible for the successful outcome of the work.

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(1) N. A. Kahn and E. A. Imbembo, "A Method of Evaluating Transition from Shear to Cleavage Failure in Ship Plate and Its Correlation with Large-Scale Plate Tests," *Welding Journal*, Vol. 27, April, 1948, pp. 169s–182s; disc., pp. 182s–185s, 216s.

(2) N. A. Kahn and E. A. Imbembo, "Notch Sensitivity of Ship Plate—Correlation of Laboratory-Scale Tests with Large-Scale Plate Tests," Symposium on Deformation of Metals as Related to Forming and Service, pp. 15–52, Am. Soc. Testing Mats. (1948). (Issued as separate publication *ASTM STP No. 87.*)

(3) N. A. Kahn and E. A. Imbembo, "Notch Sensitivity of Steel Evaluated by Tear Test," *Welding Journal*, Vol. 28, April, 1949, pp. 153s–178s.

(4) N. A. Kahn and E. A. Imbembo, "Further Study of Navy Tear Test," *Welding Journal*, Vol. 29, February, 1950, pp. 84s–96s.

DISCUSSION

MESSRS. N. A. KAHN AND E. A. IMBEMBO[1] (*presented in written form*).[2]—The discussors are happy to learn of an application of the Navy tear test other than that for which they originally developed this technique and are particularly gratified with the author's statement that "the ease and simplicity of applying the test, coupled with the large amount of information produced, point to possible great usefulness in the mechanical testing field." The discussors wish to commend the author for the excellent paper which she has presented and have but a few relatively minor comments to make.

The author indicates that, on the basis of microscopic examination, there is evidence of higher finishing temperature for the cross-rolled material. However, the microstructures do not appear to support this statement in that the cross-rolled material shows a finer and more uniform grain structure which possibly indicates a lower rather than a higher finishing temperature for the cross-rolled material. In addition, the considerably higher "as-rolled" hardness of the cross-rolled material would tend further to indicate a lower finishing temperature.

In connection with the preparation of the Navy tear specimen, the discussors wish to point out that it is their practice to finish machine the edge of the specimen opposite the notch and to locate, with suitable jigs, the loading and notch holes relative to this edge. The advantage of this procedure over that employed by the author is that the fracture length or depth of metal beneath the notch can be accurately reproduced from specimen to specimen.

The discussors wish to inquire as to whether the author has any specific plans for practical utilization of the tear test such as its incorporation in specification requirements for strip steel.

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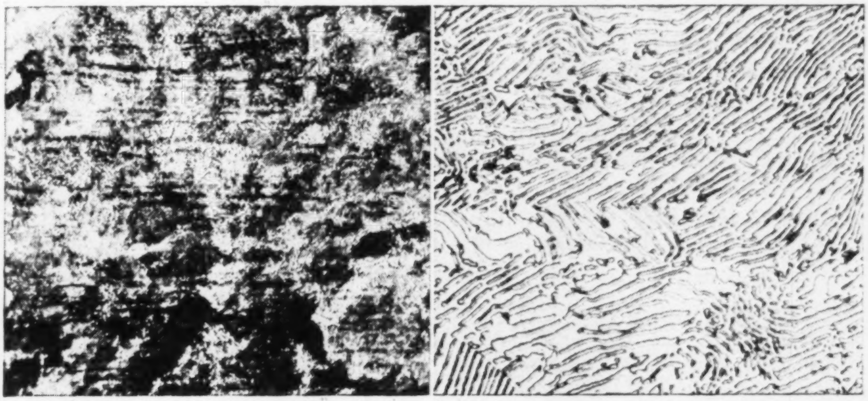
[1] Head and Supervising Physical Metallurgists, respectively, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

[2] The opinions contained herein are the private ones of the discussors and are not to be construed as reflecting the views of the Navy Department or the Naval Service at large.

MRS. HELEN D. HOOVER (*author*).—Mr. Kahn and Mr. Imbembo have questioned the statement that the microstructure showed evidence of higher finishing temperature for the cross-rolled strip. I am quite willing to agree that the finer and more even grain of the cross-rolled and its higher as-rolled hardness may be indicative of a lower finishing temperature. In support of this point of view there is also the fact, not reported in the paper, that the pearlite of the cross-rolled material, which ranged from fine to medium, was finer on the average than that of the straight-rolled, which ranged from very fine to very coarse. These variations in fineness of pearlite and hardness could have resulted from different rates of cooling after rolling, but, since the material was specially rolled in very short lengths, this did not seem to be the answer. During the study of the as-rolled material, it was observed that there was fracture and distortion of the pearlite lamillae in the straight-rolled strip and that the alignment of the dislocations pointed to a cold-rolling effect (accompanying Fig. 15). From such information as was available from the steel supplier, my conclusion was that it was likely that the reduction of the straight-rolled had been started at a temperature high enough to allow formation of very coarse pearlite, and had been completed at a temperature low enough to permit formation of very fine pearlite and also to cause cold working, while the reduction of the cross-rolled had been carried out at a temperature range somewhere inside the limits of the other, so that grain size and lamillar spacing were more even and cold working did not occur.


(*a*) Longitudinal Section. Etchant, 2 per cent Nital (× 250).

(*b*) Enlargement of Coarse Area of (*a*). Etched in 2 per cent Nital (× 1000).

FIG. 15.—Distortion of Pearlite in Straight-Rolled 1085 Strip Steel.

This question of finishing temperature is arguable, as is the question of whether or not the observed differences in as-rolled structure have notable effect on the performance of the material after hardening and tempering. Phillips and Dunkle[3] have reported that annealing low-carbon steel lowered tensile strength but did not remove the effects of previous cold rolling on the pattern produced by axial tension testing of specimens located in the five angular positions used in this application of the Navy tear test, and that earring in cupping tests was increased in the same material when cold rolling had preceded annealing. Whether carryover would be shown in tear tests of hardened-and-tempered high-carbon strip is a moot question, so that the importance of the cold working and finishing temperature in the strip used for our tests is in doubt.

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[3] Arthur Phillips and H. H. Dunkle, "Directional Properties in Rolled and Annealed Low Carbon Steel," *Transactions*, Am. Soc. Metals, Vol. 23, p. 398 (1935).

The finish machining of the edge to be notched was purely a matter of setup convenience in our work. We held the strip in a jig and cut the blanks with a fine saw, so that size variation was small, not exceeding ±0.003 in. in the nominal 2-in. fracture length. All of our fractures were "instantaneous" so that we were mainly concerned with energy absorbed to initiate fracture, and fracture length meant little. In tearing breaks, where energy to propagate fracture is of major importance, fracture length is correspondingly important and finish machining of the edge opposite the notch, as is done at the New York Naval Shipyard, has a distinct advantage. This latter method also should be used if the cutting of the blank has caused changes in the structure of the material by mechanical working or heating.

We have no current plans for using the Navy tear test for specification purposes. Opinions have been expressed that the criteria commonly used in specifying strip steel are inadequate because material well within their limits may show wide performance variation. We have detected certain corresponding differences by means of the tear test, but our data are not comprehensive enough to permit further comment.

A COMPARISON OF IMPACT TESTING MACHINES IN THE 20 TO 30 FT-LB RANGE*

By R. L. Rickett,[1] W. B. Seens,[1] R. W. Vanderbeck,[2]
C. Daniel,[3] and R. Roeloffs[1]

Reproducibility of the notch impact test from one machine to another has been a matter of some concern for a number of years (**1**).[4] This subject has become even more important as interest in notch toughness as a property of steels for various structural applications has increased. Recently several publications on the subject have appeared (**2, 3, 4**), some of which report rather wide differences in results from different machines. Because of the interest in this subject within various units of United States Steel Corp., a joint investigation was initiated in 1950 for the purpose of comparing a number of the impact testing machines used by these units.

Scope of Investigation:

It was decided to limit the investigation, initially, to tests using keyhole-notch Charpy specimens, to the energy range 20 to 30 ft-lb, and to tests made at a single temperature, 80 F. The testing program was planned, using established statistical methods, with the following objectives in mind:

1. To find even rather small differences if they exist. The original aim was to detect differences of 1.3 ft-lb or larger, but the actual results turned out to be somewhat better than this.
2. To eliminate factors that might give false indications of differences in machines.
3. To obtain results applicable to the testing of different classes of product that are most likely to be of interest.

The desired sensitivity in detecting differences among machines was obtained by testing 64 carefully selected specimens on each of the machines being compared. The effect of extraneous factors was kept to a minimum by preparing all specimens in a single shop, by random order of specimen preparation and testing, by balanced allocation of specimens to machines, and by careful attention to details of testing, including centering the specimens in the machine and making all tests at the same temperature. Generality of the results was increased by testing two general types of material having nearly the same energy absorption at 80 F but differing widely in hardness, tensile properties, and microstructure. The two classes of material selected were a structural carbon plate steel (normalized) and a hardened-and-tempered low-alloy steel (4340), specimens from several heats of each being tested.

Included in the comparison were 20 impact testing machines representing four commonly used makes. Each machine was checked in accordance with the requirements of ASTM Tentative Methods of Impact Testing of Metallic Materials (E 23 - 47 T).[5] Some of the characteristics of these machines are listed in Table I. In most instances the anvils and strikers had been modified where necessary with the aim of complying with the dimensions specified. An additional set of tests was made on one machine using 2-mm radius anvil and striker edges rather than the 1-mm radius anvil and 8-mm radius striker edges specified in Methods E 23.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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[2] Research and Development Division, U. S. Steel Corp., Pittsburgh, Pa.

[3] Engineering Statistician, New York, N. Y.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 750.

TABLE I.—RESULTS OF INSPECTION AND CALIBRATION OF TESTING MACHINES.

Machine	Make	Approx. Age, yr.	Nominal Capacity, ft-lb	Velocity, ft per sec	Anvil Radius, mm	Striker Radius, mm	Striker Width, mm	Friction and Windage Loss, ft-lb	Brake Loss, ft-lb
A	I	8	263 120	16.8 11.4	0.8–0.9 0.8–0.9	8.1 8.1	3.3 3.3	0.9 0.3	6.0 0
B	I	15	235	16.6	0.5–0.9	7.7	3.9	0.8	−6.0[a]
C	I	0–1	270 120	16.8 11.2	0.8–0.9 0.8–0.9	8.0 8.0	3.7 3.7	0.8 0.4	0 0
D	I	18	238	16.7	0.6–0.7	.8.0	4.2	3.2	−5.5[a]
E	I	2	264 115	16.8 11.2	1.0–1.1 1.0–1.1	8.0 8.0	3.6 3.6	1.7 0.7	−0.5[a] −0.5[a]
F	I	6	264 120	16.8 11.4	0.5–0.7 0.5–0.7	4.9 4.9	3.6 3.6	1.4 0.6	0.3 0
G	I	24	228	17.6	0.4–1.1	7.9	4.4	0.5	0
H-1	II	15	220	18.1	0.9–1.1	7.8	4.0	0.4	0
H-2	II	15	220	18.1	2.0	2.0	Tangent	0.4	0
I	II	8	108	12.7	0.8–1.1	8.2	3.7	0.3	0
J	II	8	109	12.8	1.1–1.2	6.3	4.3	0.3	0
K	II	17	221	18.1	1.1–1.3	6.5	3.6	0.4	0
L	II	7	110	12.8	0.9–1.0	4.9	3.9	0.2	0
M	II	4	110	12.8	1.1–1.4	6.6	4.3	0.3	0
N	II	2	110	12.8	1.3–1.4	7.9	4.1	0.2	0
O	III	4	119	17.0	0.9–1.0	8.2	4.2	0.5	0
P	III	5	121	17.0	1.0–1.1	8.2	4.2	0.6	0
Q	IV	15	162 127 47	15.4 13.6 8.1	0.7–1.0 0.7–1.0 0.7–1.0	8.1 8.1 8.1	5.3 5.3 5.3	0.4 0.2 0.1	0 0 0
R	IV	19	109 43	15.2 9.6	0.8 0.8	7.8 7.8	3.9 3.9	0.5 0.2	0 0
S	IV	24	119 46	16.0 9.9	2.3 2.3	5.9 5.9	3.3 3.3	0.4 0.1	0 0
T	IV	22	110 43	15.2 9.6	1.1 1.1	8.0 8.0	4.1 4.1	0.2 0.1	0 0

[a] Brake adds energy to pendulum.

In addition to the comparison of two types of anvil and striker on one machine, a limited number of supplementary tests were made on four machines of one make to investigate the effect of testing with or without an automatic brake, and on these four machines and on four machines of another make to compare the results of using high or low capacity ranges. In these tests, fewer specimens were used, and consequently the precision of the results is less than in the main comparison.

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[5] 1952 Book of ASTM Standards, Part 1, p. 1451, Part 2, p. 1248.

MATERIAL AND PREPARATION OF SPECIMENS

Specimens were prepared from structural carbon steel in plate form and from bars of SAE-AISI 4340. Because of the relatively large number of specimens required for each test, it was possible to include several heats of each grade and samples from different plates or bars from each heat. As a consequence, it was possible to determine the generality of the findings with respect to each grade of steel and also to obtain supplementary data on variation in notch toughness within each grade. This latter aspect, which is quite aside from the main objective of the investigation, is not considered further in this paper.

TABLE II.—COMPOSITION AND HARDNESS OF STRUCTURAL CARBON STEEL .

Heat	Ingot	Composition, per cent								Rockwell B Hardness As Normalized
		C	Mn	P	S	Si	Ni	Cr	Cu	
No. 42S257	No. 2	0.24	0.48	0.010	0.031	0.06	0.07	0.03	0.08	74.0
	No. 3	0.25	0.47	0.009	0.033	0.06	0.06	0.02	0.08	75.5
No. 27S275	No. 3	0.23	0.48	0.013	0.041	0.037	0.07	0.03	0.11	71.0
	No. 2	0.23	0.47	0.013	0.041	0.033	0.08	0.03	0.10	71.0
No. 30S258	No. 2	0.24	0.53	0.009	0.042	0.046	0.05	0.03	0.07	72.5
	No. 3	0.24	0.53	0.009	0.042	0.042	0.05	0.04	0.07	73.5
No. 31S279	No. 2	0.22	0.46	0.007	0.035	0.043	0.10	0.05	0.09	73.5
	No. 3	0.22	0.45	0.007	0.037	0.046	0.10	0.05	0.09	73.5

TABLE III.—COMPOSITION AND HARDNESS OF 4340 STEEL.

Heat	Billet	Composition, per cent									Rockwell C Hardness As Quenched and Tempered
		C	Mn	P	S	Si	Cu	Ni	Cr	Mo	
No. N43582	No. 7A	0.40	0.70	0.012	0.016	0.30	0.01	1.79	0.88	0.23	39.0
	No. 8A	0.39	0.70	0.014	0.017	0.30	0.02	1.79	0.87	0.24	39.2
No. X43578	No. 2A	0.35	0.74	0.010	0.014	0.26	0.01	1.65	0.73	0.24	37.7
	No. 5A	0.37	0.73	0.011	0.015	0.26	0.01	1.64	0.72	0.23	37.5
No. 24D187	No. 1A	0.41	0.85	0.032	0.024	0.27	0.01	1.84	0.91	0.24	39.8
	No. 1C	0.41	0.86	0.037	0.025	0.27	0.02	1.82	0.92	0.24	39.6
No. X32955	No. 4T	0.38	0.75	0.012	0.017	0.29	0.01	1.83	0.86	0.24	38.8
	No. 5T	0.39	0.75	0.012	0.016	0.28	0.01	1.84	0.83	0.24	38.7

Structural carbon steel of the composition shown in Table II was hot rolled to 0.5-in. thick plate. Each of the eight plates, two from each of four heats, was from the middle slab of the respective ingot. For this investigation, only the outer portions, 20 in. wide, from both sides of each plate, were used. These sections of plate were normalized by heating for 30 min at 1650 F and cooling in air, with the plates standing on edge to permit uniform cooling. Hardness of the normalized plate sections ranged from 71 to 75.5 Rockwell B, as given in Table II.

Eight billets from four heats of 4340 steel having the composition shown in Table III were hot rolled to $1\frac{1}{16}$-in. square bars. The first and last of seven bars from each billet were used in this investigation. The bars were heated to 1550 F in a continuous furnace, quenched in oil, and then tempered 3 hr at 1025 F. Hardness of the bars after this heat treatment was approximately 37.5 to 39.5 Rockwell C, as given in Table III.

From the bars and the sections of plate, specimen blanks approximately ½ in. square and 7 in. long were prepared. Those from the plate were cut *transversely* to the long axis of the plate and represented full plate thickness. Specimen blanks from the bar stock were prepared by quartering the bars lengthwise. These blanks were machined and ground to final cross-sectional dimensions, after which they were cut to length, three specimens being obtained from each blank. The properly identified specimens were then drilled and slotted to form the standard Charpy keyhole notch. Specimens of plate stock had the notch axis perpendicular to the original surfaces of the plate. Those from bar stock were notched so that the slot extended inward from one of the original surfaces of the bar. Specimens of each of the two grades of steel were drilled in random order. When the drills became dull, they were resharpened and checked for size of hole.

TABLE IV.—DIMENSIONS OF SPECIMENS.

	ASTM Specification	Average of 10 Specimens	Estimated Range[a]
Height, in	0.3930–0.3950	0.3943	0.3932–0.3954
Depth, in	0.3930–0.3950	0.3943	0.3934–0.3951
Reference end to center of notch, in	1.083	1.085	1.081 –1.089
Diameter of notch, in	0.079	0.0795	0.0758–0.0832
Base of notch to back of specimen, in	0.197	0.1942	0.1892–0.1992
Deviation from squareness, min	Not specified		
Carbon steel		4.6	0–19.9
4340		21.0	8.6–33.4

[a] Limits that can be expected, with 95 per cent confidence, to include 90 per cent of specimens.

Dimensions of a random group of ten specimens, five of each grade of steel, were checked with the results shown in Table IV. We consider these dimensions to be satisfactorily uniform and in reasonable agreement with ASTM requirements. Moreover, the possibility that such nonuniformity as was present might affect the results of the investigation was substantially eliminated by randomization of both specimen preparation and allocation to machines.

A sufficient number of specimens was prepared from each bar or section of plate so that two could be tested on each of the machines. It was thought that these specimens might strain-age around the notch between the time they were prepared and the time they were tested, and that the elapsed time and hence the amount of aging might differ for specimens tested on different machines. To check this possibility, half of the specimens were artificially aged for 5 hr at 210 F, whereas the others were allowed to age naturally at room temperature for the period of one to four months that elapsed between preparation and testing. It turned out that there was no difference between naturally aged and artificially aged specimens.

TESTING PROCEDURE

From the specimens representing each bar or each section of plate, one artificially aged specimen and one "unaged" specimen, each selected from the group in accordance with a table of random numbers, were assigned to each machine. The 64 specimens thus selected for each machine were arranged in random sequence for testing, so that there would be no consistent relationship between specimen source and order of testing.

The temperature of testing was specified as 80 ± 1 F to avoid the rather sizable difference in impact values that might result if tests were made at different temperatures. Data from another source indicate that for the carbon plate steel a difference of 10 F in temperature of test, in the range used, would result in approximately 2 ft-lb difference in impact value. The specified test temperature is well above the expected transition temperature of the steels used and for the carbon plate steel corresponded to approximately 30 per cent of granular fracture area. Fracture of the heat-treated 4340 steel was entirely fibrous. Most of the specimens were dry when tested, but in a few instances they were warmed to the desired temperature in water and tested while still wet.

TABLE V.—RESULTS OF MACHINE COMPARISON (64 TESTS PER MACHINE).

	Machine	Capacity Used, ft-lb	Variance	Standard Deviation	Average Charpy Value,[a] ft-lb
Make I	A	263	1.69	1.30	27.48 ± 0.33
	B	235	0.86	0.93	26.07 ± 0.24
	C	270	1.22	1.11	25.86 ± 0.28
	D	238	0.62	0.79	25.15 ± 0.20
	E	264	0.67	0.82	25.04 ± 0.21
	F	264	0.44	0.66	24.88 ± 0.17
	G	228	1.73	1.31	24.57 ± 0.33
	Average		1.03	1.02	25.58
Make II	H-1	220	0.56	0.75	25.48 ± 0.19
	I	108	0.62	0.79	25.44 ± 0.20
	J	109	0.60	0.77	25.16 ± 0.20
	H-2	220	0.60	0.77	25.12 ± 0.20
	K	221	0.40	0.64	25.02 ± 0.16
	L	110	0.86	0.93	24.49 ± 0.24
	M	110	0.58	0.76	24.39 ± 0.19
	N	110	0.71	0.84	24.11 ± 0.21
	Average		0.62	0.79	24.90
Make III	O	119	1.13	1.06	25.24 ± 0.27
	P	121	0.46	0.68	24.29 ± 0.17
	Average		0.79	0.89	24.77
Make IV	Q	162	0.83	0.91	25.14 ± 0.23
	R	109	0.74	0.86	24.17 ± 0.22
	S	119	0.37	0.61	23.90 ± 0.16
	T	110	1.25	1.12	23.59 ± 0.29
	Average		0.80	0.89	24.20

Grand Average: Charpy impact value........ 24.96 ft-lb
Variance..................... 0.86
Standard Deviation......... 0.93

[a] 95 per cent confidence limits for the average are listed.

In testing, the indicator was set to read zero on the scale after a free swing of the pendulum, and the zero position was checked frequently during testing. Test data were recorded in most instances to the nearest 0.5 ft-lb, but on a few machines the scale could not be read closer than 1 ft-lb. Values were reported as read from the indicator, without any correction based on calibration of the machines.

COMPARISON OF MACHINES

The tests that were made yield information on two separate aspects of the comparative behavior of impact testing machines of the group investigated. The first of these is the relative level of impact values and the second is the degree of consistency, or reproducibility, of the various machines.

The range in average impact values for the 20 machines is approximately 4 ft-lb, as indicated by the data in Table V. These impact data are the averages of actual test values, without any corrections based on calibration of the machines. The observed averages are, of course, subject to some uncertainty as are all experimental measurements. The extent of this uncertainty can be estimated from the data and expressed as limits within which the "long-range" average of a much larger number of similar tests can be expected to lie. Such predicted long-range limits for the average corresponding to each machine are listed in the last column of Table V. These limits are based on the probability that they would fail to include the long-range average for only one machine out of twenty (95 per cent confidence interval). It is evident from these limits that the relative level for each machine has been established within approximately ± $\frac{1}{4}$ ft-lb in the range of 20 to 30 ft-lb covered by these tests.

Because of the close limits within which the relative levels of these machines have been established, it is possible to differentiate between machines for which the observed averages differ by as little as 0.32 ft-lb. The actual difference between any such pair of machines can be considered to be the observed difference ± 0.32 ft-lb. These statements, again, are based on the chance that only one time in twenty will the conclusion be in error.

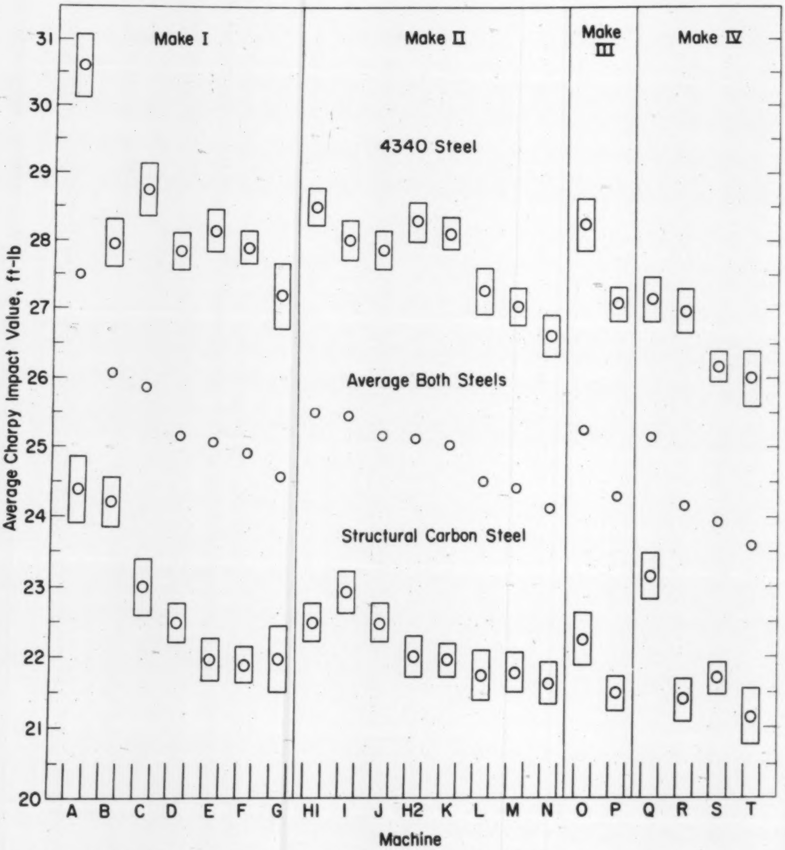

FIG. 1.—Comparison of Machines Based on Averages of All Tests, on Results for 4340, and on Results for Structural Carbon Steel.

The comparisons shown in Table V are based on average values for an equal number of specimens of two widely different types of steel. The same *general* trends exist for the averages based on either type of steel individually, as shown in Fig. 1, where the machines are arranged in the same order as in Table V and the over-all averages for the machines are also shown. There are, however, minor differences, and a statistical test was used to determine whether these differences are significant. If the differences among machines were the same for one steel as for another, each of the upper points in Fig. 1 (for 4340 steel) should be above the corresponding lower point (for the carbon steel) by the same amount, within experimental error. It was found the the observed differences vary more than would be expected from the possible errors in measuring the averages. It is concluded, therefore, that the relative levels of impact values for the different machines may depend on the type of steel tested and are not the same in all instances when testing a relatively soft steel, such as the plate steel used here, as when testing a harder, stronger steel, such as hardened-and-tempered 4340 steel.

Further analysis of the data revealed that although the relative impact energy levels for different machines are affected by differences between the two types of steel tested, presumably because of their wide differences in properties, other than notch toughness, these relative levels are not affected by such minor differences as exist from heat to heat or within heats of the same general type of steel.

The machines were also found to differ in the consistency, or reproducibility, of their results as indicated by the values of standard deviation listed in Table V. These values are a measure of the reproducibility of duplicate tests, the expected *average* difference for any pair of duplicate specimens being 1.13 times the standard deviation given for that machine. This expected average difference ranges from 0.7 ft-lb for the most consistent machine (*S*), to 1.5 ft-lb for the least consistent (*G*).

The standard deviations in Table V include both the variability of the respective machines from test to test and the average variation from specimen to specimen within each lot of steel tested. Because of the random selection of specimens and the rather large number of pairs tested on each machine, the specimen variability should average nearly the same for each set of tests. The standard deviation values, therefore, can be considered a valid indication of *relative* variability of the individual machines. They cannot, however, be used to predict the average difference between duplicate specimens of steels less uniform than those used in this investigation.

COMPARISON OF MACHINE MAKES

Because the makes of machines differ in design and construction, the data were examined to see if there are consistent differences in results for different makes.

As indicated in Table V, the observed average level is highest for Make I machines as a group (25.6 ft-lb) and lowest for the Make IV machines (24.2 ft-lb) with the other makes being intermediate. Whether such differences are generally true for other machines of the makes represented is not certain, but there is a rather strong probability (confidence level of approximately 90 per cent) that they are. This suggests that there may be some difference in construction or design that results in lower values when Make IV machines are used than when, for example, tests are made using machines of Make I. What these differences are has not yet been established.

There are also differences in apparent level of absorbed energy among the machines of any one make, and these differences are greater than the differences in average level from one make to another. Of the three makes for which several machines are represented, the greatest variation is in the group of Make I machines and the least variation is in those of Make II.

The degree of reproducibility of testing is poorest for the Make I machines as a group (average standard deviation 1.02) and is best for the Make II machines (average standard deviation 0.79). The Make I machines also have the widest range of reproducibility, values of standard deviation extending from 0.66 to 1.31, whereas the machines of Make II are the most consistent in this respect, values of standard deviation for these machines ranging from 0.64 to 0.93.

CORRECTIONS BASED ON CALIBRATION OF MACHINES

Some of the variation in relative level of impact energy observed in this investigation may be due to errors in the energy values that were indicated by the machines. Accurate calibration of the machines would, of course, eliminate this source of variation. It is doubtful, however, that methods of calibration now commonly used are sufficiently accurate to detect small errors in measuring the amount of energy absorbed in breaking a specimen.

Despite limitations, and some degree of ambiguity, in established calibration procedure, an effort was made to calibrate each of the machines used in this investigation. With the indicator first set to read zero on a free swing under the same conditions as used in testing, the potential energy of the pendulum was determined at several positions corresponding to known scale readings. These potential energy values were computed from the calibration weight of the pendulum, measured as described in ASTM Tentative Methods E 23 – 47 T, and the elevation of the middle of the striking edge above the lowest point of swing. When, as in this investigation, the energy absorbed in a test is only a small portion of the total scale range, the total friction and windage losses should be only slightly less in a test than in a free swing, and the difference can be neglected. The energy absorbed by the specimen in a test then becomes the difference in potential energy of the pendulum at the two positions corresponding, respectively, to zero and to the observed value on the scale.

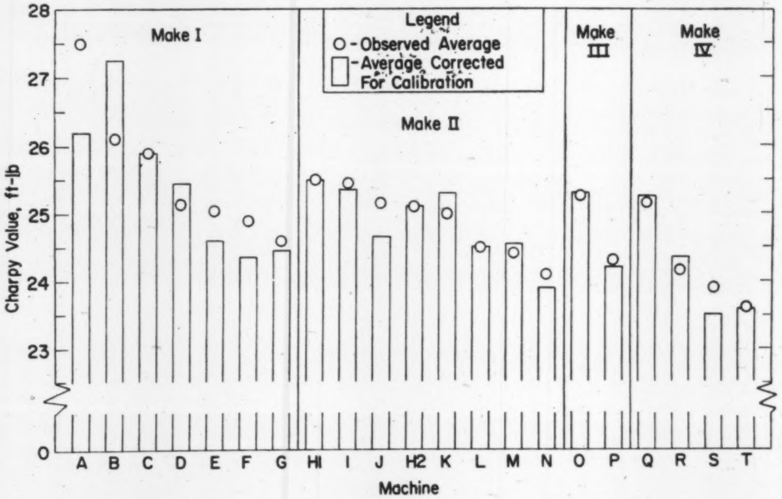

FIG. 2.—Observed Averages for Machines Corrected on Basis of Reported Calibrations.

As a basis for computing such differences, the potential energy measurements made at 4 to 10 scale positions for each machine were used to calculate a 'best" straight line for that machine by established statistical methods. In view of possible slight departures from linearity of scale and magnitude of the possible experimental errors in measurement of pendulum weight and height, it is doubtful whether the machine calibrations that were determined in this manner can be considered accurate to better than ±0.5 ft-lb at the 25 ft-lb energy level.

The averages for all of the machines, corrected on the basis of the calibration just described, are shown in Fig. 2. The corrections are small for most machines and they do not improve the uniformity of the results. The corrections do change the relative order of the machines in some instances, but it is not known whether these changes are significant.

TABLE VI.—CORRELATION BY RANK BETWEEN DEFORMATION OF SPECIMENS BY ANVILS AND TOTAL ABSORBED ENERGY (STRUCTURAL CARBON STEEL).

Machine	Rank with Respect to Total Energy Absorbed (Highest listed first)	Relative Order of Specimen Deformation (Highest = 1)
A	1 (24.38 ft-lb)	2
B	2	1
Q	3	6
C	4	3
I	5	15
H-1	6	4
D	7.5	5
J	7.5	16
O	9	8
H-2	10	12
G	11	20
E	12.5	19
K	12.5	7
F	14	18
M	15	9
L	16	11
S	17	21
N	18	14
P	19	17
R	20	13
T	21 (21.17 ft-lb)	10

EFFECT OF TESTING VARIABLES

A limited number of observations or tests were made to investigate the possible effect that differences in condition of the machines or their method of operation may have had on the average Charpy energy level obtained. These included an examination of the amount of deformation of the specimens by the anvils, the effect of anvil radius, the effect of machine capacity, and the effect of the automatic brake on some of the machines of Make I.

Specimen Deformation:

Considerable difference was noted in depth of anvil impressions in specimens tested on different machines. To determine whether the amount of such deformation correlated with total energy absorbed, a representative specimen of structural carbon steel tested on each machine was compared with corresponding specimens from each of the other machines by each of six observers. Based on these ratings, which agreed fairly well, it was possible to rank the machines in relative order of the amount of deformation produced and to compare this order with that of the average indicated energy values for the same grade of steel. The resulting rankings, presented in Table VI, indicate a definite trend in the direction of greater absorbed energy for the more severely deformed specimens. Statistical analysis of these data indicated a highly significant correlation (probability of chance occurrence less than 0.01).

The amount of deformation of these specimens did not appear to be closely related to the reported values of anvil radius given in Table I. These values, however, represent a single measurement made on a wax impression of each anvil and do not necessarily represent the extreme variations.

Radius of Anvils and Striker:

Included in the data of Table V are the results of two sets of tests made on Machine H, both with new anvils and striker edges. In one set of tests (H-1), anvils having a 1-mm radius and a striking edge with a radius of 8 mm and a width of 4-mm, both as specified in Methods E 23 – 47 T, were used. In the other set of tests (H-2), an edge radius of 2-mm was used for both anvils and striking edge, in accordance with usual practice for this machine. The results of this second set of tests averaged 0.36 ft-lb (0.36 ± 0.32 ft-lb) lower than the first set. Although only one machine was used in this comparison, the results indicate that when the anvils and machine are in good condition a considerable variation in edge-radius of the anvils and in contour of the striking edge can be expected to have only a slight effect on test results in the range of 20 to 30 ft-lb.

Capacity Effect:

The machines of Make I, which as a group were highest in energy level, were used at high capacity and most of the machines of Make IV, which were the lowest group, were, with one exception, used at a considerably lower capacity level. This led to an exploratory test to determine whether, for each of these groups, a change in the capacity of the machines would affect the results. In these tests four of the Make I machines were used at two capacity levels, nominally 264 ft-lb and 120 ft-lb. These machines were also compared with and without brakes, as described later. In these tests a total of 16 specimens were broken on each machine. The four machines of Make IV were compared at two lower capacity levels, approximately 120 and 45 ft-lb. In these tests 12 specimens in all were broken on each machine.

TABLE VII.—ESTIMATED CAPACITY EFFECT, MAKE I.[a]

Machine	4340 Steel			Carbon Plate Steel		
	High Capacity	Low Capacity	Difference	High Capacity	Low Capacity	Difference
A	32.25	30.0	2.25 ± 1.41	24.50	24.50	0.0 ± 1.41
C	25.25	24.62	0.63 ± 1.41	19.75	20.38	−0.63 ± 1.41
F	30.88	28.13	2.75 ± 1.41	23.50	23.38	0.12 ± 1.41
E	26.50	26.88	−0.38 ± 1.41	22.00	22.25	−0.25 ± 1.41

[a] Each entry, in ft-lb, is average of 4 observations.

No *consistent* effect of machine capacity was found. Of the four machines of Make I, two appear to have a significantly higher average impact level when used at high capacity than at low, but only for the 4340 steel, as shown in Table VII. Because of limited data, the differences for the Make IV machines were not broken down by steels. For these machines, no definite effect of capacity was found (Table VIII).

It is evident from these results that more data are needed before any final conclusions can be drawn regarding the effect of machine capacity. It is equally evident that if such conclusions are to be at all general in their application, tests must be made on a number of machines and different types of steel must be included to encompass a range of hardness and impact energy levels.

TABLE VIII.—ESTIMATED CAPACITY EFFECT, MAKE IV.[a]

Machine	High Capacity	Low Capacity	Difference with 0.95 Confidence Limits
Q	29.67	31.00	−1.33 ± 1.45
R	23.38	23.04	0.34 ± 1.45
S	25.95	24.75	1.20 ± 1.45
T	24.38	24.50	−0.12 ± 1.45

[a] Each entry, in ft-lb, is average of 6 observations.

Effect of Automatic Brake:

To determine whether the larger range in level of impact values for the Make I machines and their lower average reproducibility were associated with the automatic brake with which some of these machines are equipped, tests were made on four machines with and without the brake in operation. These were the same tests that were used to investigate the effect of capacity, the tests being balanced with respect to these two variables. On each of the four machines used, eight specimens were broken with the brake in operation and eight with the brake removed. For each condition, the scale was adjusted to read zero on a free swing of the pendulum, or the reading was corrected for displacement of the zero. As indicated by the values for brake loss in Table I, considerable adjustment was required in some instances.

TABLE IX.—EFFECT OF AUTOMATIC BRAKE, MAKE I.

Machine	Brake On, ft-lb	Brake Off, ft-lb	Difference, ft-lb
A	27.4	28.3	0.9 ± 1.0
C	22.6	22.4	−0.2 ± 1.0
F	26.4	26.6	0.2 ± 1.0
E	24.4	24.4	0 ± 1.0

[a] Each value given is average of 8 specimens.

For these conditions, no significant difference was found between the results of tests made with the brake and those made without the brake. The level of impact values was the same in both instances, within the precision of the measurements, as indicated in Table IX. This was true for both steels and for all four machines. Reproducibility of testing under the two sets of conditions also was essentially alike, the standard deviation being 0.90 with brake and 1.08 without brake. These values correspond to expected average differences between duplicate specimens of 1.02 ft-lb and 1.21 ft-lb, respectively.

SUMMARY

From the results of this comparison of machines, using keyhole-notch specimens and restricted to tests in the 20 to 30 ft-lb range and at approximately room temperature, the following conclusions may be drawn:

1. Averages of observed impact values for the 20 machines investigated differ by a maximum of approximately 4 ft-lb. Many of the machines differ by a smaller, but statistically significant, amount, differences greater than 0.32 ft-lb in the average of 64 tests on each machine being considered significant.

2. There are variations in impact energy level among the machines of each make and also in average level for the different makes, Make I being highest and Make IV lowest.

3. Although the general order of impact energy level for the machines is the same for structural carbon steel as for harder and stronger 4340, there are minor differences in relative order for the two grades that are statistically significant.

4. There are statistically significant differences among the machines in precision or reproducibility of results.

5. Similarly, there are significant differences in average reproducibility of results among the makes, Make I machines, in general, giving the least reproducible results and the machines of Make II the most reproducible results.

6. The application of corrections based on calibration of the machines does not reduce the variation in impact energy level among machines. It does change the relative order of the machines in some instances, but these changes in order may not be significant.

7. One apparent cause of differences in relative energy level among machines is the amount of specimen deformation produced by the anvils, the indicated energy levels being generally higher for the machines that caused the most severe deformation.

8. Based on tests on only one machine, use of anvils and striker as specified in ASTM Methods E 23 – 47 T gives results that are only slightly higher than when 2-mm edge radius anvils and striker

are used, the difference being 0.36 ± 0.32 ft-lb.

9. There is some indication that the capacity range used may, in some instances, affect the energy level, but more data are needed before definite conclusions can be drawn.

10. No evidence was found in these tests that use of the automatic brake on one make of machines affects the results. Under the conditions used in these tests, the effect, if present, must be small.

Acknowledgments:

The authors wish to thank all of the many individuals in various plants and laboratories of U.S. Steel Corporation and its divisions who participated in these tests or were instrumental in supplying material to be tested.

References

(1) N. A. Kahn and E. A. Imbembo, "Reproducibility of the Single-Blow Charpy Notched-Bar Test," ASTM Bulletin, No. 146, May, 1947, p. 66.

(2) Metallurgical and Research Committee, Forging Manufacturers Assn., "How Accurate is the Impact Test?" *Metal Progress,* Vol. 60, Dec., 1950, pp. 69–71, 176.

(3) C. A. Penrose, "How Good is the Charpy Impact Test?" *Iron Age,* Vol. 170, No. 11 Sept. 11, 1952, pp. 143–147.

(4) H. L. Fry, "An Inquiry into the Reproducibility of Impact Test Results," ASTM Bulletin, No. 187, Jan., 1953, p. 61.

DISCUSSION

MR. H. L. FRY.[1]—I would like to congratulate the authors of this paper on the very carefully planned and executed piece of work, one that I think is concisely and well reported.

It is quite apparent from the paper that the variability among the machines is real. It cannot be accounted for on the basis of specimen preparation or any of the other factors that are usually held to account for this type of variability. At first sight the amount of variability does not seem extreme, but it should be noted that the maximum variation of 4 ft-lb from the highest to the lowest machine actually amounts to about 16 per cent of the average of all the machines. That seems to me to be rather more than the variability to be expected in any kind of precision test.

Of course, it might be pointed out that the amount of variability will be important only in borderline cases, but in the business of making acceptance tests, we who have to do this are living on the borderline all the time, because of the fact that people who write specifications for materials generally tend to ask for as much as possible from a material—in some cases a little more.

I was interested in the comparison of the amount of deformation of the specimens with the general order of magnitude of the impact results, and it seems to me that the findings there suggest that the amount of energy used in deforming the ends of the specimen may be larger than we think. It may be actually on the same general order of magnitude as the amount of energy required to fracture the specimens—at this level, at least. I feel that future research should try to develop the reasons for this variability and should make some attempt to account for the absorbed energy—that is, the energy absorbed from the pendulum in the various ways in which it is absorbed—and try to get an idea of how much is going into the fracture of the specimen and how much is going into extraneous work. I feel that along that line we can eventually arrive at some answer to the entire problem.

MR. NOAH A. KAHN.[2]—I would like to call the authors' attention to an observation which we made in connection with a study of the reproducibility of the Charpy notched-bar test involving twelve laboratories. While the reproducibility obtained at relatively high energy levels was adequate, the reproducibility of some machines was poor when testing specimens of low energy absorption corresponding to a predominantly brittle behavior with energy values of under 10 ft-lb. This, in part, was due to a tendency for the broken halves of the specimen to jam in the testing machine. It would be very interesting and informative if the authors initiated a supplementary investigation involving a study of impact testing machines in connection with a material and test conditions which would result in an energy absorption in the range of approximately 3 to 12-ft lb. Such a study is considered important and timely in view of the increased emphasis which is being placed on transition temperature definition based on energy levels under 20 ft-lb. Consideration should be given to the inclusion of the V-notch Charpy specimen as well.

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[2] Principal Metallurgist, New York Naval Shipyard, Brooklyn, N. Y.

MR. DAVID E. DRISCOLL[3] (*by letter*).—The authors are to be congratulated on their manner of selecting two steels with the same energy values but differing widely in hardness, tensile properties, and microstructure, and also on the care used in heat treatment and machining of specimens; but the results would have been more convincing if a greater percentage of the specimens had received a dimensional check and the actual spread in results obtained were recorded in addition to the average. Table IV of the paper shows that in checking only 10 of the 1280 specimens, the average depth below the notch is 0.1942 in. One then wonders what the actual spread was and what the spread would have been if a greater percentage had been checked. To say that the dimensions were satisfactorily uniform and in reasonable agreement with ASTM standards is not borne out by the results obtained.

In the summary the authors state that differences greater than 0.32 ft-lb in the average of 64 tests are significant, yet the text states that (*a*) it is doubtful whether the machine calibrations could be considered accurate to better than ±0.5 ft-lb and that (*b*) test data were recorded in most cases to the nearest 0.5 ft-lb, but in some cases could not be read closer than 1.0 ft-lb.

Taking into consideration the fact that these machines have not been calibrated or thoroughly checked, I wonder if the authors realize how well these machines have checked. If the high machine and the two low machines in Fig. 1 (4340 steel) are eliminated, the remaining 18 machines show a spread of ±1.4 ft-lb or ±5.0 per cent. Tension testing machines are considered very reliable, but I doubt if the tension test results obtainable on machines in eighteen different plants would produce results as uniform.

MR. G. C. RIEGEL[4] (*by letter*).—Industry owes a vote of thanks to the authors for the light which they have shed on the moot question of the calibration of the so-called impact testing machines for breaking notched bars.

Outside of differences in geometry of specimens, particularly the variations in acuity of notches, the greatest variable between specimens has been the physical nonhomogeneity of the material tested. The authors have taken considerable pains to select their materials but failed to show by photomicrographs what differences may have existed around the notches of the specimens which had the greatest scatter.

It is our opinion that hot-rolled, normalized, low-carbon steel is subject to considerable nonuniformity in the distribution of ferrite and pearlite. Such was evident in the report of N. A. Kahn and E. A. Imbembo to which the present authors made reference. It is a factor of too great importance to be dismissed lightly.

While the quenched-and-tempered constructional alloy steels are not as subject to heterogeneous physical conditions as are the ferrite-rich, low-carbon steels, especially the "non-killed" types, yet "banding" or ferrite streaks in the nickel-rich steels are frequently encountered. The inception of the notch fracture in a ferrite band gives a different propagation than in a carbide-rich band. Fracture initiation at the root of a notch in a specimen which has been carburized a few tenths of a thousandth during hardening and tempered at less than 450 F will, on No. 2345 or No. 4340 reduce the ft-lb values from an average of 16.0 to an average of 3.0.

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[4] Chief Metallurgist, Caterpillar Tractor Co., Peoria, Ill.

Nonuniformity as measured by hardness is a very significant factor and should not be permitted to spread with any greater latitude on specimens used for calibration of impact testing machines than one would permit for calibrating hardness testing machines. There can be easily as much as 8.0 ft-lb difference in energy absorption between 36.0 and 41.0 Rockwell C hardness on the No. 4340 specimens described by the authors. A similar statement may be made with respect to the Rockwell B scale hardness.

Strength, which resists the deformation of bending when a notched-bar specimen is broken by impact, registers its contribution to the energy values absorbed by significant amounts until brittleness becomes dominant. Strength, which is most readily measured by indentation-hardness, must be uniform in material used for calibration purposes.

The variability of different machines in producing scatter of energy values on uniform material is clearly brought out by the authors and is one of the worst faults of this type of testing machine.

It would be a valuable contribution to industry if the National Bureau of Standards would evaluate and publish the factors of accuracy which must be fulfilled by impact machines for reproducibility and calibration for this type of testing. The differences in test machines should never be so great that the differences in tests results could isolate them. We should certainly condemn tension testing machines if they behaved so poorly.

MESSRS. R. L. RICKETT, W. B. SEENS, R. W. VANDERBECK, C. DANIEL, AND R. ROELOFFS (*authors' closure*).—We agree with Mr. Fry that the variation in machines, although small, is greater in a few instances than may sometimes be desirable. We plan to continue our efforts to find the cause of the observed differences, and if possible to reduce them. As mentioned by Mr. Fry, our results indicate that the energy used in deforming the specimen where it makes contact with the anvil edges may be one cause of variation among machines. As yet, however, we have no information on the amount of energy thus absorbed. Some of the machines used in our investigation produced very little deformation of this kind, but the impact energy values were only slightly lower than for machines that produced much more severe deformation. Because of this, we doubt very much that the energy absorbed in deformation of the specimens by the anvils was of the same order of magnitude as that required to break the specimen, as suggested by Mr. Fry.

The testing of predominantly brittle specimens as advocated by Mr. Kahn was not included in our investigation because we felt that the actual energy levels reported in such instances would be of little interest or value except in very specialized applications. It is true that the likelihood of jamming might be increased, but we believe that when serious jamming occurs it can be recognized and the results of such tests discarded. Keyhole-notch energy values in the range 10–20 ft-lb are of definite interest but often are within the "transition range," in which case they are likely to be so variable that a very large number of specimens must be tested if differences among machines are to be detected. If only a small number of specimens are tested, apparent differences in machines may be due only to chance variations in the specimens themselves. In fact, we wonder whether the poor reproducibility mentioned by Mr. Kahn was not actually due to the wide scatter that normally results when testing keyhole Charpy specimens within the transition range.

In additional tests that are planned we do expect to include V-notch Charpy specimens as suggested by Mr. Kahn.

The specimen dimensions, mentioned by Mr. Driscoll, were checked only as a matter of record. Although only 10 randomly selected specimens were measured, it is possible to estimate from these measurements the variation to be expected in the entire lot of over 1300 specimens tested. The limits thus computed are, however, somewhat broader than would probably be found if a larger number of specimens was measured. Whether or not the estimated ranges, which are included in Table IV of the paper, are satisfactorily in agreement with ASTM standards is largely a matter of opinion because limits are specified for only a few of the dimensions.

In planning the tests, precautions were taken to minimize the effect of such variations as those in specimen dimensions mentioned by Mr. Driscoll and in hardness and microstructure mentioned by Mr. Riegel. Each lot of material tested was chosen so as to be reasonably uniform, and specimens from each lot were tested on each machine. All specimens were prepared in a single shop in random sequence, and those from each lot were assigned to the several machines in accordance with accepted statistical procedure. In view of these precautions, we believe that each group of 64 specimens was, as a whole, nearly identical with any other group even though there were undoubtedly differences among corresponding specimens in the various groups. The relatively small average difference between duplicate specimens indicates that the effect of variation in material and method of preparation must have been small.

Mr. Driscoll implies that the significance of small differences in averages of sets of 64 tests is questionable because the machines could not be read more closely than 0.5 or 1.0 ft-lb. He overlooks the fact that these average values are much more precise than the individual measurements. The machines were calibrated, but as stated in the paper we are not certain that there were no errors in the calibration. Such errors, if present, may be responsible for some of the apparent differences among machines.

We cannot agree with Mr. Driscoll's statement that if a similar careful comparison were made of tension testing machines the results would differ by as much as ±5 per cent.

We greatly appreciate all of the comments that have been offered and hope that all those interested in this field will join in an attempt to resolve the many questions that still have not been answered.

DELAYED YIELDING IN ANNEALED LOW-CARBON STEEL UNDER COMPRESSION IMPACT*

By J. E. Johnson,[1] D. S. Wood,[1] and D. S. Clark[1]

Synopsis

The delay time for the initiation of yielding in an annealed low-carbon steel is investigated experimentally under longitudinal compression impact. Delay times in the range from 40 microseconds to 1.5 milliseconds are determined as a function of the initial impact stress at temperatures of 73, 150, and 250 F. The stress *versus* delay time relation at 73 F is found to be in good agreement with an extrapolation from previous data at longer delay times and lower stresses obtained by rapid-load tension tests on the same material. The initiation of yielding is found to be accompanied by a decrease in stress, the magnitude of which does not vary systematically with the initial impact stress.

Previous investigation **(1,2)**[2] has shown that the delay times for the initiation of yielding in an annealed mild steel depends upon the applied stress and temperature. Heretofore, specimens were subjected to a rapidly applied constant tensile stress in which the stress rise time (5 to 10 milliseconds) was small compared to the total time duration of the test. The purpose of the present experiment is to measure delay times of less than 5 milliseconds as a function of stress and temperature.

An experimental method has now been developed by which delay times of less than 5 milliseconds can be detected. The technique used to accomplish this is as follows: A constant compression stress is applied to one end of a long cylindrical specimen by longitudinal impact with a heat-treated bar which remains elastic during the test. The compression stress is maintained at the end of the specimen for a period of time corresponding to the time for an elastic wave to propagate twice the length of the heat-treated bar. If yielding occurs before this time, plastic deformation is initiated at the impacted end of the specimen, resulting in a decrease of stress. Thus, a delay time of less than the time required for an elastic wave to travel twice the length of the heat-treated bar can be determined.

Experimental Method

The experimental method used in this investigation is shown schematically in Fig. 1. A free cylindrical bar, A, traveling with a velocity, V_0, impacts a stationary anvil bar of the same diameter consisting of four sections, B, C, D, and

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 764.

E. One end of section *E* rests on a fixed base. Joints between *B*, *C*, *D*, and *E* are lapped plane so that longitudinal compression waves are transmitted without disturbance. The yield strength of bars *A*, *B*, and *D* is higher than the stress imposed by the impact; hence, these bars behave elastically during the test.

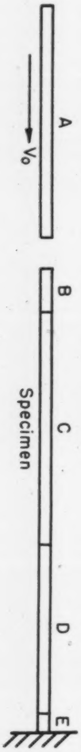

FIG. 1.—Schematic Diagram of Experimental Method.

The bar *C* is the test specimen, whose upper yield stress is exceeded during impact. Bar *E* is a material with a very low yield strength and serves to absorb most of the impact energy by plastic deformation.

Bar *A* moving with a velocity V_0 strikes bar *B*, and elastic compression waves of equal magnitude are propagated through each bar. The magnitude of these waves is determined by satisfying the continuity of stress and particle velocity at the interface *A*-*B* and is given by:

$$\sigma_A = \sigma_B = \frac{V_0}{\dfrac{1}{\rho_A c_A} + \dfrac{1}{\rho_B c_B}} \quad \ldots\ldots (1)$$

where:

σ = the impact stress,
ρ = the mass density of the material,
c = the velocity of propagation, and

subscripts refer to the particular bar. One compression wave travels through the bar *A* and reflects from the free surface as a tension wave of equal magnitude. The other compression wave travels through *B*, *C*, and *D* as though the bars were continuous, since the values of ρ and c are the same for each bar. When this wave reaches section *E*, plastic deformation is initiated in *E* because of the lower yield strength. The length of bar *A* is made less than the total length of *B*, *C*, and *D*; hence, the time required for the first reflection wave from *E* to reach the interface *B*-*C* is greater than the time required for the reflecting wave in bar *A* to reach the interface *B*-*C*. Thus, the interface *B*-*C* is always unloaded by the tension wave in bar *A*; consequently, all complex reflections from bar *E* need not be considered.

At a later time after impact, corresponding to the delay time for the material, plastic deformation is initiated in the specimen at the interface *B*-*C*. This yielding is accompanied by a decrease in stress, giving rise to a tension unloading wave of low stress.

The stress-time relation in the specimen can be determined by experimental means; hence, the initial stress, the drop in stress due to yielding, and the delay time can be measured. The strain-time relation can also be determined in a similar manner in which the initial

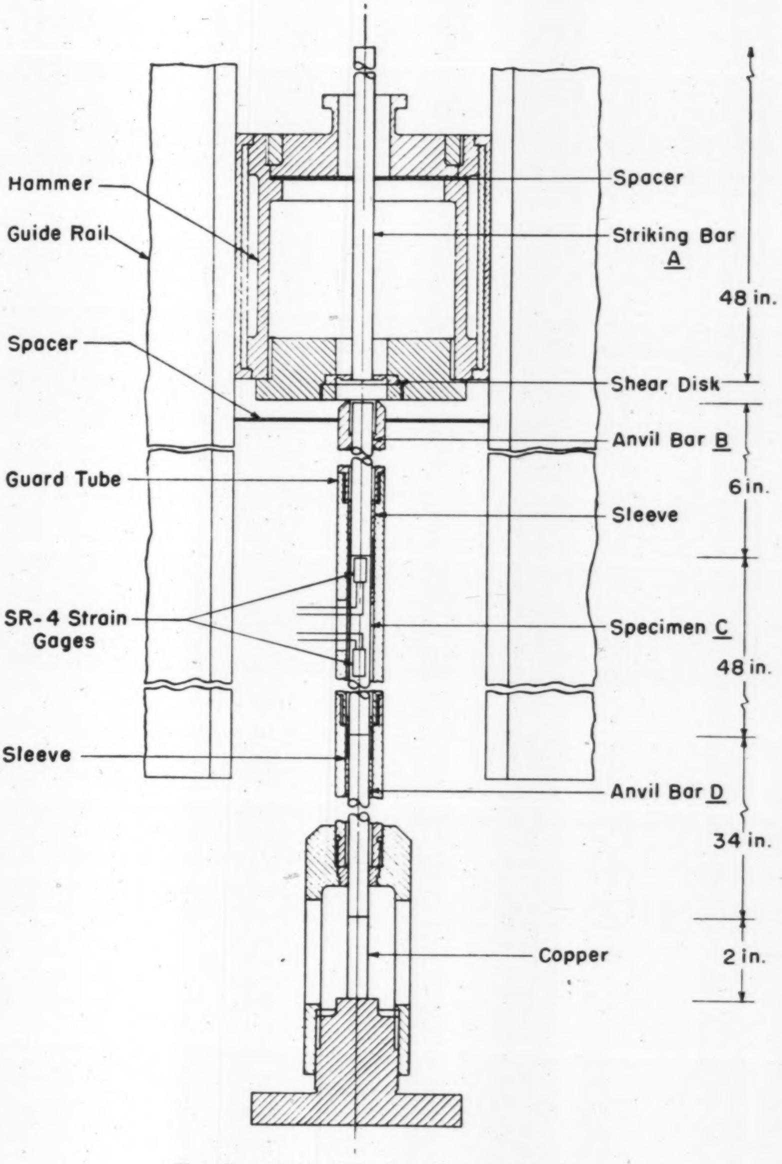
Hammer
Guide Rail
Spacer
Guard Tube
SR-4 Strain Gages
Sleeve
Spacer
Striking Bar A
48 in.
Shear Disk
Anvil Bar B
6 in.
Sleeve
Specimen C
48 in.
Anvil Bar D
34 in.
Copper
2 in.

FIG. 2.—Section Drawing of Impact Machine.

elastic strain and the delay time can be measured.

EQUIPMENT

Vertical Impact Machine:

The tests were made with a vertical impact machine, a section drawing of which is shown in Fig. 2. A guard tube is held vertically in a central position between the rails of the machine by means of an expendable spacer. The anvil bar sections shown in Fig. 1 are placed within the guard tube and centrally located by means of sleeves. Since some misalignment between the anvil bar sections and the striking bar will occur, the top end of section *B* is provided with a convex spherical surface, having a 19 in. radius, to prevent initial impact with the corner of the bar.

The hammer, which slides on the two vertical rails, is provided with a central hole through which the guard tube passes. Twenty rubber bands $\frac{3}{8}$ in. thick and 1 in. wide, attached to the hammer and the frame of the machine, serve as a means for accelerating the hammer to the desired impact velocity. The hammer is raised by a lifting mechanism to a height at which the extension of the rubber bands will give the desired velocity. The bar *A* is held centrally in the hammer by means of a shear disk shrunk onto the bar and clamped in the hammer. The shear disk strikes the top of the guard tube just prior to the impact of bar *A* on bar *B* in such a manner that bar *A* is released from the shear disk and impacts bar *B* without disturbance from the shear disk. The shear disk fails along a circumferential groove whose diameter is slightly larger than the guard tube. This allows the hammer to continue downward, passing around the guard tube. It is decelerated by means of four vertical friction brakes.

Tests above room temperature are performed by placing a split tube heater containing ten furnace elements along the bars *B*, *C*, and *D*. The guard tube, specimen, and anvil bar sections are heated to the desired test temperature. The heater is removed just before the test to prevent obstruction of the hammer motion.

Recording System:

The velocity of the hammer is determined by measuring the time to travel between three fixed points near the position of impact. As the hammer passes each of the fixed points, it closes an electric circuit, causing a discontinuity in the sweep on an oscilloscope screen. A timing trace from an audio-oscillator is also placed on the screen of the oscilloscope. The oscilloscope screen is photographed and the number of cycles between the discontinuities is counted, thus giving the time required for the hammer to travel the known distance between contact points.

The strain in the specimen is measured as a function of time during impact with the use of SR-4 resistance-sensitive wire strain gages cemented to the specimen. The strain gage for measuring strain *versus* time is connected in a voltage dividing circuit which is energized by batteries. Means for introducing known resistance changes are provided in the circuit for calibration of the records. The strain is recorded by a suitable cathode-ray oscilloscope system employing a single sweep, linear time base.

The response of the recording system is determined by impressing a 5000 cps square wave having a rise time of less than $\frac{1}{2}$ microsecond on the strain gage circuit and recording in the usual manner. The rise time of this square wave on the oscilloscope screen is 40 microseconds. This minimum rise time of the recording system imposes a lower limit on the delay times which can be measured.

The temperature of the specimen in the tests at elevated temperatures is determined at each end and the center by means of three copper-constantan thermocouples and a Leeds & Northrup portable potentiometer with suitable switching arrangements for the various thermocouples. The heaters are controlled by a Leeds & Northrup Micromax controller in conjunction with a thermocouple placed on the outer surface of the guard tube. The outer surface of the guard tube is maintained at a different temperature than that of the specimen. This temperature is adjusted to produce the desired specimen temperature. This provides a better means of controlling the specimen temperature within the desired range than can be obtained by placing the controlling thermocouple on the specimen.

The tests at elevated temperatures are performed by heating the guard tube and specimen to a temperature higher than the desired temperature. When the heaters are removed before the test in order to prevent obstruction of the hammer motion, the specimen temperature decreases at a rate of 2 F per min, and the hammer is released when the desired temperature is reached. In this manner, the specimen temperature at impact is maintained to within ± 2F of the desired temperature. The temperature gradient from the top to the middle of the specimen is less than 3 F, and the temperature gradient from the middle to the bottom of the specimen is less than 20 F. Since plastic deformation occurs only in the top 2 in. of the specimen, the temperature gradient can be neglected.

MATERIAL TESTED AND TEST SPECIMENS

The specimens used in this investigation were machined from $\frac{5}{8}$-in. diameter, hot-rolled bars from a single billet of Heat No. 32882, Columbia Steel Co., Torrance, Calif. The analysis as given by the mill is as follows:

Carbon	0.17 per cent
Manganese	0.39 per cent
Phosphorus	0.017 per cent
Sulfur	0.040 per cent

The material is the same as that used in a previous investigation (2).

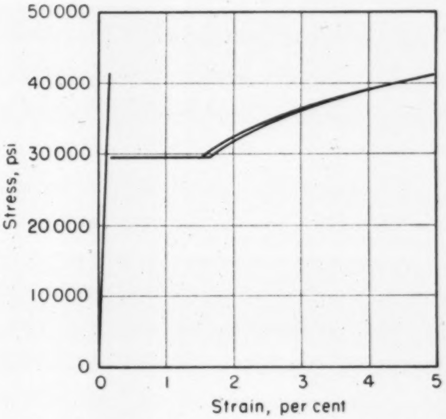

FIG. 3.—Static Stress *versus* Strain, 73 F.

The test specimens were centerless ground to a diameter of 0.5000 ± 0.0005 in. over their length of 48 in., and the ends were ground plane. After grinding, the specimens were annealed in a controlled atmosphere, neutral packed at 1600 F for 1 hr, and furnace cooled to room temperature. The ends of the specimens were lapped perpendicular to the longitudinal axis after heat treatment to assure uniform contact with the anvil sections and thus prevent wave reflections from the contact surfaces. Shorter test specimens were cut from the longer specimens and lapped to a specimen length of $11\frac{3}{4}$ in.

EXPERIMENTAL RESULTS

Static Tension Tests:

Two static tension tests were made at 73 F. The static stress-strain relations for strains up to 5 per cent are shown in Fig. 3. Specimens for these tests were of the same type used in a previous investigation (2) and were machined from impact specimens chosen at random. The procedure of this previous investigation was followed. The tests were performed on a 150,000-lb Olsen Universal Testing Machine having a least reading of 1 lb (corresponding to a stress of 19 psi in the gage section). The machine was recently calibrated and showed an error of less than 0.75 per cent. A comparison was made between the static stress-strain curves obtained in this investigation and the curves obtained in a previous investigation (2). The comparison indicates that the stress-strain relations of the specimens used in both investigations are the same.

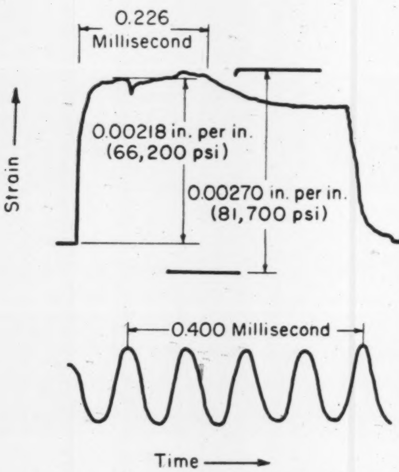

FIG. 4.—Tracing of a Record of Strain *versus* Time 2¼ in. from End of Specimen Tested at 73 F.

Determination of the Velocities of Elastic Waves:

The velocities of elastic waves in the specimen, anvil bar sections, and striking bar were determined by measuring the resonance frequency of the bars in longitudinal vibration. The bar or specimen was rigidly clamped at its quarter points, which were the nodes for the second mode of vibration. A permanent magnet loudspeaker was placed at each end of the bar with the moving coil cemented to the end of the bar. One of the loudspeakers was driven by an audio-oscillator, while the other loudspeaker was used as a pickup whose output was displayed on an oscilloscope screen. The resonance frequency of the bar in longitudinal vibration corresponded to the maximum amplitude of the detector signal on the oscilloscope screen.

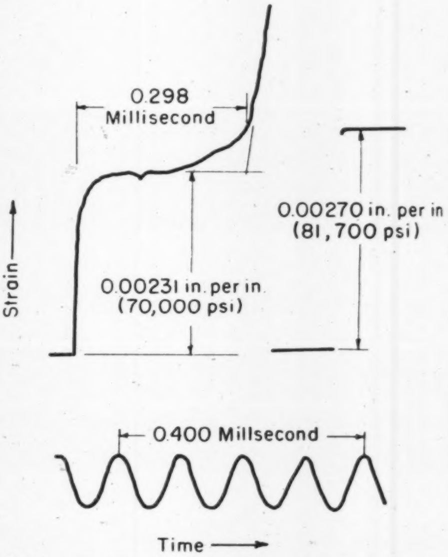

FIG. 5.—Tracing of a Record of Strain *versus* Time $\frac{1}{16}$ in. from End of Specimen Tested at 73 F.

The velocity of propagation of the elastic wave c_0 is:

$$c_0 = \frac{2fl}{n} \quad \text{(2)}$$

where:

f = the resonance frequency of longitudinal vibration,

l = the length of the test bar, and

n = the mode of longitudinal vibration.

Young's modulus is also computed from:

$$E = \rho c_0^2 \quad \text{(3)}$$

where ρ is the mass density of the material.

The average velocity of elastic waves in the test material, obtained from six tests, is 16,900 ft per sec. The average velocity of elastic waves in the striking bar and anvil bar sections, obtained from two tests, is 16,700 ft per sec. The deviation of values of elastic wave velocities from the mean value is less than 0.4 per cent. It is estimated that the accuracy in the determination of elastic wave velocities is within ±0.6 per cent.

Young's modulus of each material is computed from Eq 3, using ρ equal to 7.34 lb sec^2 per in.[4] Young's modulus of the test material is 30.3×10^6 psi, and the modulus of the anvil bar sections is 29.6×10^6 psi.

Compression Impact Tests:

A series of tests was made at each of three temperatures, 73 F, 150 F, and 250 F. Impact velocities were chosen to obtain a uniform distribution of test points in the plot of delay time *versus* stress. The minimum value of delay time which could be determined was limited by the rise time of the oscilloscope. Since the stress at the end of the specimen is maintained for only a limited time in these tests, the delay times which can be measured are necessarily less than this time limit. This limit was the time for an elastic wave to travel twice the length of the striking bar. However, the limit could be extended by performing repeated impacts on the same specimen until yielding occurred. A previous investigation (3) has shown that a specimen yields when the cumulative time at stress is approximately equal to the normal delay time, provided the aging period between impacts is shorter than the critical recovery time. This method could be used only at 73 F.

The strain in the specimen was measured as a function of time at either one of two positions along the specimen. In some tests, the strain was measured $2\frac{1}{4}$ in. below the top end of the specimen, a position at which only elastic strain occurred. From these measurements, the decrease in stress due to yielding and the delay time could be determined. In some tests, the strain was measured $\frac{1}{16}$ in. below the top end of the specimen. The latter measurements provided a better means for determining the delay time than the former.

A tracing of a record of strain *versus* time for a position $2\frac{1}{4}$ in. from the end of the specimen tested at 73 F is shown in Fig. 4. This record shows that the strain in the specimen at this position remains elastic, since the trace deflection returns to zero at the end of the record. The record also shows the decrease in strain associated with the unloading wave which accompanies yielding.

A tracing of a record of strain *versus* time for a position $\frac{1}{16}$ in. from the end of the specimen tested at 73 F is shown in Fig. 5. This is similar to the strain-time records obtained from the rapid-load tests of a previous investigation (2) and clearly shows the phenomenon of delayed yield. The recording system was arranged to provide a relatively high sensitivity for the strain measurement so that the initial elastic strain at impact could be determined accurately. The delay time is determined from the record in the manner indicated in Fig. 5.

In some tests the time at which yielding occurred was difficult to determine because the decrease in stress accompanying yielding was so small. For this reason, some of the tests at 73 F and all the tests at 150 and 250 F were performed by using a strain gage placed $\frac{1}{16}$ in. from the end of the specimen. This provides a much more sensitive means of determining the time at which yielding begins and, hence, a better means of determining the delay time. This can be seen by comparing Figs. 4 and 5. The decrease in stress accompanying yielding indicated in Fig. 4 is

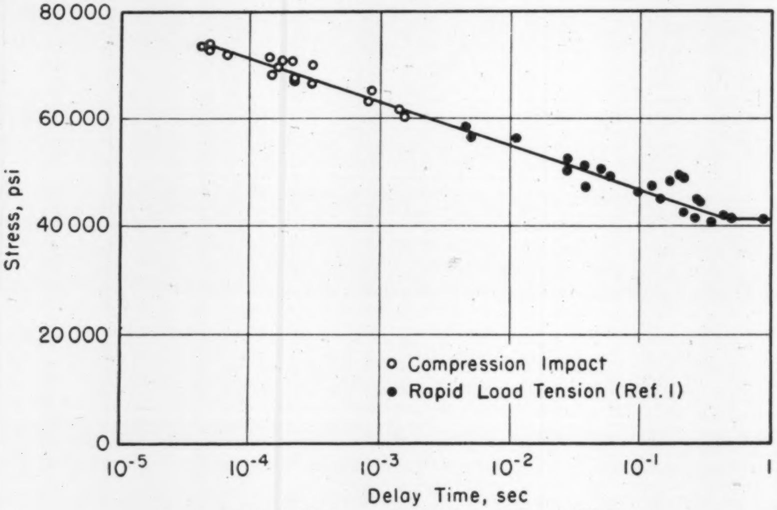

FIG. 6.—Delay Time as a Function of Stress, 73 F.

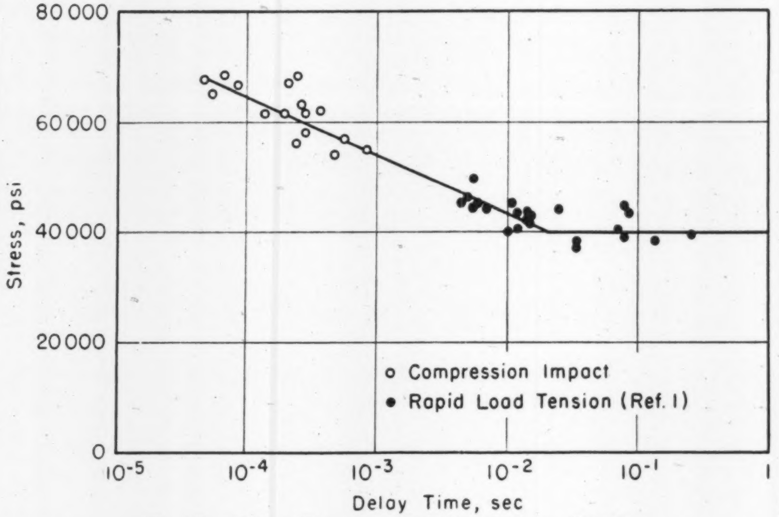

FIG. 7.—Delay Time as a Function of Stress, 150 F.

small, so that the time at which the decrease begins is difficult to determine accurately, whereas the beginning of plastic strain in Fig. 5 is very prominent and provides a more satisfactory means of determining the time at which yielding begins.

The stress at impact for all the tests is computed from the velocity of impact by means of Eq 1. In those tests at 73 F in which high sensitivity strain-time records were obtained, the stress at impact could also be accurately determined from the records. The stresses measured from the strain-time records are within ±1.2 per cent of the stresses calculated from the impact velocity.

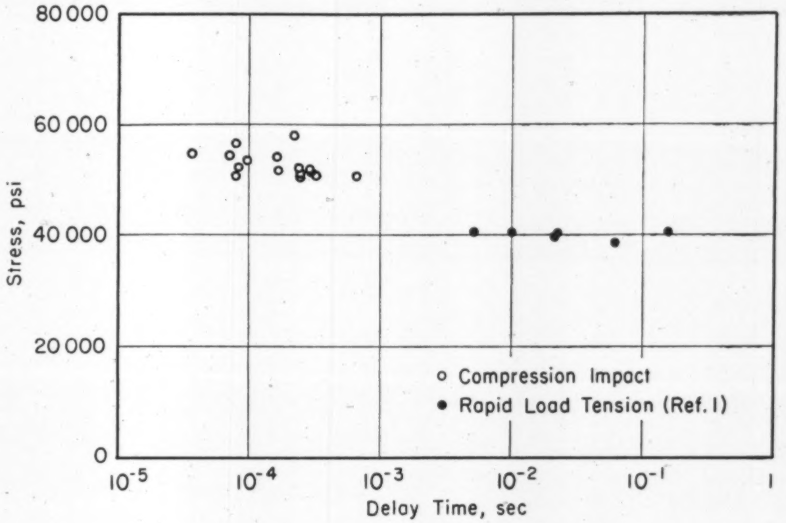

FIG. 8.—Delay Time as a Function of Stress, 250 F.

The points representing stress *versus* log delay time are plotted in Figs. 6, 7, and 8. Data from a previous investigation (2) are also plotted for comparison. The lines drawn to represent the data are determined in the same manner as in the previous work. The lines of negative slope may be represented by a relation of the form:

$$t = t_0 e^{-\sigma/\sigma_0}$$

The lines of zero slope may be represented by:

$$\sigma = \bar{\sigma}$$

The numerical values of the constants, t_0, σ_0, and $\bar{\sigma}$ are given in Table I. The values of t_0 and σ_0 cannot be obtained for the tests made at 250 F because the data are not sufficient to be represented by a line.

The line representing the data obtained at a temperature of 73 F in the compression tests of this investigation is the same as the extrapolated line representing the data secured in the rapid-load tension tests of a previous investigation (2) at this same temperature for the same material.

TABLE I.—EMPIRICAL CONSTANTS TO FIT THE RELATIONS $t = t_0 e^{-\sigma/\sigma_0}$ AND $\sigma = \bar{\sigma}$ TO THE EXPERIMENTAL DATA.

Temperature, deg Fahr	t_0, sec	σ_0, 10^3 psi	$\bar{\sigma}$, 10^3 psi
73.............	3.34×10^4	3.64	41.0
150.............	16.8	4.55	40.7
250.............	..	..	40.0

A comparison of the static stress-strain curves obtained previously (2) with the curves of the present investigation indicates that the material used in the two investigations possesses nearly identical stress-strain relations. Hence, it may be concluded that the phenomenon of delayed yielding is essentially the same for either tensile or compressive loading. The relations between stress and delay time presented in Figs. 6 and 7 show that the slope of the line representing the data at 150 F is greater than the slope of the line representing the data at 73 F. This result is in contrast with results of previous work in which it was found that the slopes of the lines at 73 F and −75 F were very nearly the same.

The decrease in stress accompanying yielding may be determined from the strain-time records obtained at a position $2\frac{1}{4}$ in. from the end of the specimen in some tests at 73 F. The decrease in stress ranges from 6100 to 17,200 psi and is independent of the initial impact stress in the range of stresses studied in this investigation. Thus the decrease in stress accompanying yielding under impact conditions appears to be somewhat random in nature.

SUMMARY AND CONCLUSIONS

Delay times for yield in annealed low-carbon steel in compression impact exist and are of the same nature as found in rapid-load tension tests. The relation between stress and delay time determined in compression impact tests at 73 F agrees with the extrapolated relation obtained from rapid-load tension tests on the same material. Hence, the determination of delay times in compression impact tests is comparable with the results of rapid-load tension tests. Compression impact tests are useful for determining delay times of smaller magnitude than can be determined from rapid-load tension tests. This technique is also used to measure delay times at 150 and 250 F.

The decrease in stress occurring in the specimen at yielding is found to be of random nature and cannot be correlated with the value of initial impact stresses.

Acknowledgment:

This investigation was conducted at the California Institute of Technology under the sponsorship of the Office of Naval Research. The authors express their appreciation to this agency for their support of the work.

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DISCUSSION

MESSRS. G. R. IRWIN AND J. M. KRAFFT.[1]—The authors have presented a significant addition to their studies of delayed yield phenomena. Their present work demonstrates that compression-loading delay times exist and exhibit the same dependence on stress and temperature as tensile-loading delay times. We wish to submit two questions and a comment on this paper.

Consider, as appears to be the case, that the plastic deformation is confined to a region not far from the first loaded end of the specimen; such behavior is to be expected in view of the low plastic wave velocity in material with yield point characteristics. The remainder of the bar specimen remains elastic. Strains measured on the elastic portion of the bar (Fig. 4) are proportional then to the load supported by the region in plastic deformation. The corresponding plastic strain history is measured on the sample of specimen covered by the gages $\frac{1}{16}$ in. from the end. Within this approximation we then note from Fig. 4 that the stress supported by the specimen exhibits a tendency to drift upward prior to the onset of gross yielding. Since this increase in stress could act to decrease the delay time with respect to that which would be observed under truly constant stress, some comment by the authors on this point would be appreciated.

The dip in this stress (Fig. 4) at about 80 microseconds might indicate a momentary yield or loss of stress-supporting ability somewhat before the gross yielding at 226 microseconds. This dip appears to be characteristic of both the records shown. It would be interesting to resolve this with a recording system having a minimum response time less than 40 microseconds. Is it possible that elastic behavior terminates here rather than at 226 microseconds?

Finally, we note that the friction between the elastic anvil bar B and specimen C provides some biaxial restraint to plastic deformation in the specimen material close to this boundary. This protects the end of the bar from yielding much as the oversize ends of tension specimens protect adjacent specimen material from yielding. Compression impact experiments at our laboratory suggest absence of this frictional restraint greatly reduces or eliminates the yield delay time.

MR. W. H. HOPPMANN II.[2]—I am not fully aware of the significance of this time delay. Perhaps it is a characteristic of material, or perhaps it arises as a result of technique, or perhaps a combination of both. I have devoted considerable time to this type of testing, and I am also very much interested in carefully defining the analytical background for such testing.

According to the authors, we could go from stress to strain by Hooke's law. That is the ground upon which one may readily raise a question immediately. What is this Hooke's law that relates the stress to strain under these conditions of loading? It puts us in a very embarrassing position, because we do not have the basis for examination that we have in a static test, where we very slowly apply a load and measure the total load, measure the deformation under the load, and then relate the two. Here is essentially a dynamic condition, and in order to answer questions fairly concerning a dynamic condition we must solve a set of equations based on a theoretical definition of the problem. That is our difficulty.

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[1] Naval Research Laboratory, Washington, D. C.

[2] Associate Professor of Mechanical Engineering, The Johns Hopkins University, Baltimore, Md.

I can sympathize completely with the authors because I do not have any good answer for that phase of the problem. But I think one should at all times keep in the foreground this essential difficulty, because the answer to it will throw a light upon the nature and reactions of the materials and the methods of test and their relations one to the other.

For example, about ten years ago Von Kármán wrote on the velocity question concerning the impact test. He assumed a statical relation between stress and strain and set up the nonlinear differential equation for the description of the plastic phase in a bar; but in all fairness to von Kármán we know that he very clearly said and underlined: "This analysis is based on the engineering stress-strain relation from a statical machine and I am not so sure that this assumption is permissible, but if it is, then we can proceed with the analysis in this fashion."

So I caution very strongly against such assumptions as that we can use a static law for relating stress and strain, because when we come to the plastic region especially, this is the highly questionable part.

With regard to the experimental portion, there are questions regarding the action of the strain gage in the plastic region. Very little is said about the strain gage: as to its action under highly loaded areas of the bar, as to whether it is properly recording strain on the end of the bar where a spherical head was put in partly for reasons of expedience. I think that we should give close consideration to these phases of the question, even if we have to make experiments for fifty years, rather than go off too freely with regard to new phenomena that we may find—such as delayed time and so on.

Finally, if the delayed time is to have meaning as far as materials engineers are concerned, what is its general significance and, more important, what is its real nature?

MESSRS. J. E. JOHNSON, D. S. WOOD, AND DONALD S. CLARK (*authors' closure*).—Messrs. Irwin and Krafft have raised two questions concerning the details of the measurements made and one concerning the technique. We believe that the apparent tendency for the stress to drift upward during the delay time which is observed in the test record reproduced in Fig. 4 is of little significance. This apparent drift may be largely attributed to characteristics of the oscilloscope employed (9-in. RCA, Type 327A). Thus we have observed that sweep traces made above or below the horizontal center line of the cathode-ray tube when no vertical impact signal is applied are always curved slightly with the concave side toward the center line. This is due to the curvature of the face of the cathode-ray tube and possibly some interaction between the horizontal and vertical deflection systems of the oscilloscope.

The small dip in the records at about 80 microseconds may be attributed to minor disturbances due to the experimental arrangement. The small dip was observed in some other records but not all of them. We feel that it probably represents a small compression pulse generated in the striking bar, A, when the shear disk is stripped from the bar by the guard tube prior to impact. Such a compression pulse would travel upward in the striking bar, reflect from the free end, and return as a tension pulse. Then if the impact takes place during the round trip travel time of this pulse or some multiple of that time, the returning tension pulse will pass on down to the specimen and be recorded in the manner observed.

Radial friction forces at the interface between the anvil bar, *B*, and the specimen, *C*, are not developed, we believe, until plastic deformation of the specimen begins. During the delay time while both bars are reacting elastically their Poisson's expansions are equal since both bars have the same elastic properties. Thus friction of this type is present only as a result of plastic deformation in the specimens. Hence we feel that the delay time is not affected, although the rate and final amount of plastic deformation which occurs after the delay time may be decreased by this friction effect.

Preliminary tests were made during this investigation in which the impact took place directly on the specimen. These tests always resulted in yielding with no delay time. This was felt to be due to the unavoidable stress concentration at the impact surface. For this reason the arrangement was changed to that indicated in the paper, thus substantially eliminating elastic stress concentrations at the end of the specimen.

The authors agree with Mr. Hoppmann that in the range of plastic strains the stress-strain behavior of a material under dynamic load conditions may be quite different from the behavior under static conditions. However, we believe that our experimental results show that no appreciable plastic strains occur during the delay time for yielding. Thus the interpretation of the tests in terms of a delay time for yielding only involves the question of the validity of Hooke's law under dynamic loads. These experiments as well as those of other investigators indicate that Hooke's law is valid under dynamic loads, with the understanding that the inertia of the material is properly accounted for by the appropriate equations of motion. For example, elastic strain produced by the impact may be predicted as a function of the impact velocity on the assumption that Hooke's law holds and using the equations of motion. Since we measured both the strain and impact velocity a check on the theory was possible, and this showed good agreement between theory and experiment. Also the theoretical time for elastic waves to traverse twice the length of the striking bar, which depends upon the same assumptions, checked very well with experimental values.

Finally, we feel that the delay time for yielding is a real characteristic of the material and not a characteristic of the technique of testing. The references given in the paper refer to studies of the delay time which were made using a very different technique of testing. Since the two techniques give consistant results, it seems difficult to maintain that the observed delay for yielding is a characteristic of the technique. This does not exclude the possibility that the delay time is also a function of other variables than those investigated, such as the type of a multiaxial stress state or the magnitude of stress gradients. Furthermore, the delay times measured refer to a time at substantially constant stress in the sense that the rise time of the stress is relatively short compared to the delay time. If a stress which varied continuously with time up to the instant at which yielding begins were applied, a delay time, as the term is used in the paper, could not be defined. However, in our view none of these possibilities invalidate the concept of the delay time for yielding at constant stress as a characteristic property of the material.

THE EFFECT OF RANGE OF STRESS ON FATIGUE STRENGTH OF NOTCHED AND UNNOTCHED SAE 4340 STEEL IN BENDING AND TORSION*

By W. N. Findley,[1] F. C. Mergen,[2] and A. H. Rosenberg[3]

Synopsis

Fatigue data are presented for SAE 4340 steel hardened to Rockwell C 38. The data include results of tests at different values of mean stress in bending and in torsion for both notched and unnotched specimens.

Since some of the stresses employed were such as produced yielding, the stress distribution resulting from yielding was analyzed to determine the actual stresses employed at the start of the test. The permanence of these stresses was also examined.

The mechanism of failure and effect of notches are discussed, as well as combined stress theories of failure and a proposed theory for the effect of mean and maximum stress on the fatigue strength.

In this paper the term "range of stress" is used in the broadest sense, as employed by Smith (**1**),[4] for example, to describe in a general way a relationship between the maximum and minimum stress in a cycle of stress. A complete description of a sinusoidal cycle of stress requires two algebraic quantities, such as maximum and minimum stress, alternating stress amplitude and mean stress, range ratio and maximum stress, alternating stress amplitude and maximum stress, alternating stress amplitude and minimum stress, and so on.

Previous Work

The effect of range of stress has been investigated and discussed widely. The problem was reviewed by Peterson (**2**) in 1937 and existing data interpreted by Smith (**1**) in 1942. Some of the subsequent investigations (**3-8**) were reviewed and Smith's interpretation for tensile mean stresses brought up to date by Schwartz (**9**) in 1948. Other studies of the effect of range of stress are found in references (**10-19**).

Various empirical relations have been proposed to describe the observed effect of mean stresses in fatigue. Among these are the modified Goodman law (**20**), the Gerber parabola (**21**), the Soderberg linear relation (**22**), Smith's equation for brittle metals (**1**), and Seliger's parameter (**23**).

For torsion fatigue of unnotched duc-

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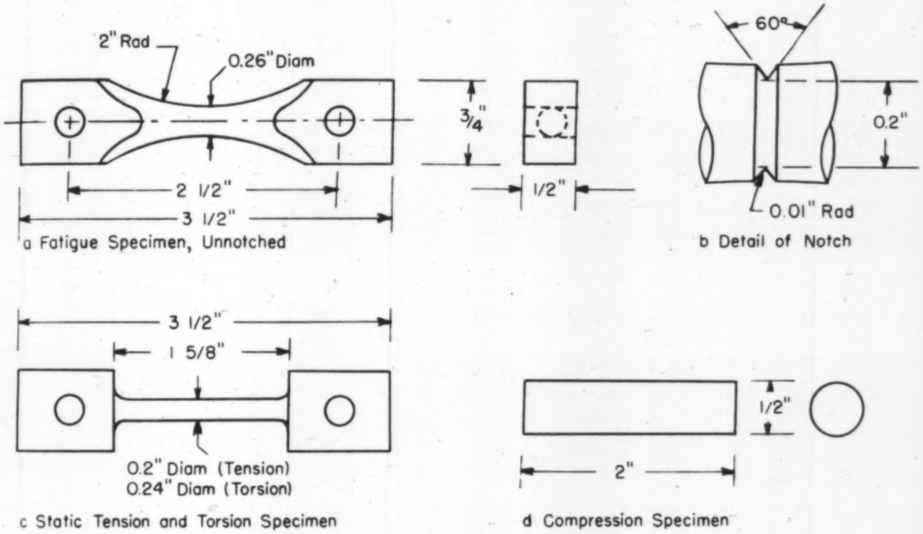
[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p 783.

tile metal specimens, Smith (1) observed that the fatigue strength was nearly independent of the mean stress when the maximum stress was less than 0.8 of the yield strength. For notched specimens in torsion and axially loaded specimens, he observed that the fatigue strength decreased with increasing mean stress.

It has been observed (17, 19) that the fatigue notch factor was influenced by the mean stress as well as the theoretical stress concentration factor.



a Fatigue Specimen, Unnotched

b Detail of Notch

c Static Tension and Torsion Specimen

d Compression Specimen

FIG. 1.—Specimens.

A theory of fatigue failure proposed by Orowan (26) was based on the concept that fracture starts when a critical stress is reached in an inhomogeneity or when the numerical sum of both positive and negative plastic strains reaches a critical value. This theory predicted that the fatigue strength was independent of the mean stress as long as the elastic limit was not exceeded. The same conclusion was reached by Freudenthal (27) in a theory of fatigue failure based on the statistical aspect of fatigue.

Recently Findley (24, 25) suggested that the fatigue strength might be independent of mean stress except as the maximum stress caused changes in the structure of the material, either by elastic or plastic deformation.

Material and Test Specimens

The SAE 4340 steel for this investigation was manufactured by Rotary Electric Steel Co. from heat No. 16632 in the form of 1-in. diameter hot rolled and annealed bars of aircraft quality. The following certified chemical analysis was furnished with the material: C, 0.40; Mn, 0.74; Ph, 0.015; S, 0.030; Si, 0.30; Cr, 0.82; Ni, 1.79; Mo, 0.26. The McQuaid-Ehn grain size was 7 to 8. The stock was heat treated in 1-ft lengths at Hamilton Standard Div. of United Aircraft Corp. in the following manner: normalized from 1650 F, quenched in oil from 1550 to 1600 F, heated for 2 hr at 1040 to 1050 F, and cooled at 200 F per hour to 500 F. This treatment produced a Rockwell C hardness of 36.7 to 39.0 and the structure of tempered martensite throughout the bar.

The dimensions of test specimens employed are shown in Fig. 1. The reduced sections of the unnotched specimens were ground to size and then polished successively with 1, 0, 00, and 000 emery paper using a $\frac{3}{4}$-in. diam bobbin rotating at 1750 rpm while the specimen was rotated at 725 rpm. The notched specimen was the same as the unnotched except for the circumferential groove machined at the minimum section.

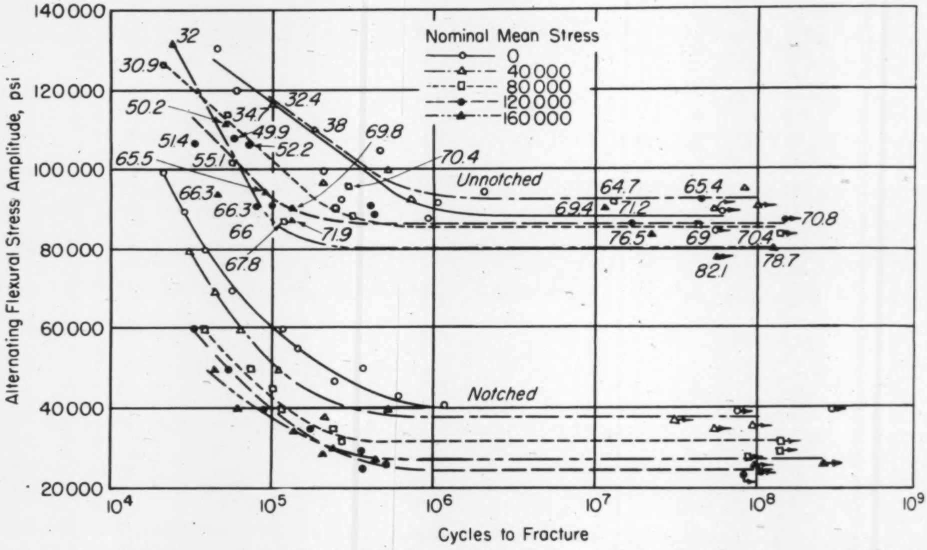


FIG. 2.—*S-N* Diagrams for Bending Fatigue of Notched and Unnotched Specimens at Different Mean Stresses. Numbers at points indicate mean stresses corrected for yielding. Alternating stresses for unnotched specimens are corrected for yielding.

The root of the V in the notched specimens was polished by rotating in the notch a 0.020 in. diam soft copper wire with Nortons grain 240 abrasive and oil until all tool marks were removed. Polishing was then continued with carborundum grain FFF. The stress concentration factor given by Neuber (**28**) for the unnotched specimens was practically unity, but for the notched specimens it was about 2.6 for bending and 1.65 for torsion.

The static tension and torsion specimens were machined as shown in Fig. 1 from bending fatigue specimens which had run out.

FATIGUE TESTS

The apparatus employed has been described (**29**) previously, but the testing technique used was different. In the present tests the deflections to be imposed on each specimen were determined by applying in succession a dead load that would produce the minimum stress and one that would produce the maximum stress and noting the deflection in each case. The fatigue machine was then adjusted to reproduce these deflections.

Application of the largest calibration loads caused the specimens to take a permanent set in many instances, the amount of which was a function of the load and time under load. The amount of the permanent set was recorded and was found to be less than 2 per cent of the deflection for all but about the three highest stressed specimens in each series of tests. The yielding, and permanent set which followed, in specimens loaded beyond the yield point caused both mean and alternating stresses to be different from the nominal values calculated from the loads. These stresses were corrected as described later for tests of unnotched specimens.

One specimen tested in torsion was accidently subjected to an excessively large amplitude of deflection. It endured 1500 cycles, but during this time (1 min) it became so hot that a dark blue oxide formed on the surface. The heat was evidently generated by the energy dissipated by a large hysteresis loop resulting from yield at both extremes of the stress cycle.

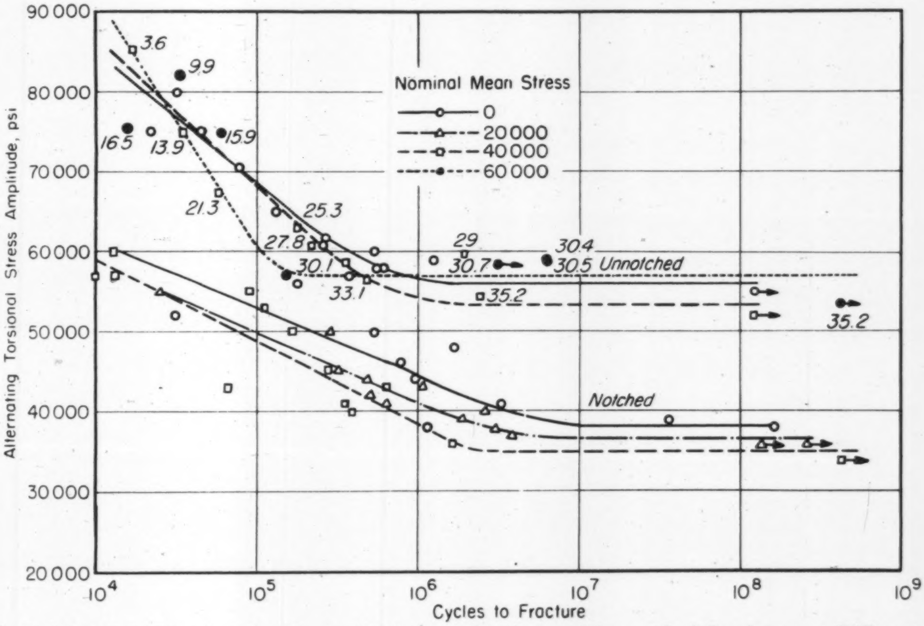


FIG. 3.—*S-N* Diagrams for Torsion Fatigue of Notched and Unnotched Specimens at Different Mean Stresses. Numbers at points indicate mean stresses corrected for yielding. Alternating stresses for unnotched specimens are corrected for yielding.

In Figs. 2 and 3 are shown the *S-N* diagrams for fatigue tests in bending[5] and torsion of notched and unnotched specimens. Each *S-N* diagram was drawn separately by inspection in order to represent the trend of the data. The values of alternating stresses shown have been corrected as described later. As has been observed before, there is less scatter in results from the notched than the unnotched specimens. Some of the scatter in results of tests of unnotched specimens is the result of variations in the material. As noted above, the Rockwell C hardness of the specimens varied from 36.7 to 39.0. A comparison of hardness with the fatigue results, however, disclosed no consistent trend. Because of a shortage of material, three notched torsion specimens were obtained by notching previously tested unnotched specimens which had not fractured. The stress to which material at the root of the notch was subjected in the first test was considered to be too low to have damaged the material.

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[5] The bending fatigue tests were first described in the Hamilton Standard Propellers Report *HSP-509*, March 21, 1946.

Results of the tests of the specimens in the notched form were normal.

Another factor which indicates a variation in the material is the fact that the unnotched fatigue specimens tested in bending did not fail consistently in the highest stress position of the specimen. In fact, some specimens fractured as much as 0.11 in. from the center of the specimen, where the stress was 4 less than at the center. Analysis showed that the standard deviation in location of fracture relative to the center of the specimen was 0.0506 in. for unnotched specimens tested in bending. These observations indicated some nonuniformity of the material. No correction of stresses was made for variations in location of origin of fracture. Variation in material was not so apparent in results of notched specimens or torsion specimens since the fractures all started in the notch in notched specimens and the origin of the fracture was not so apparent in torsion tests.

FATIGUE FRACTURES

Examination of the fractured specimens disclosed the following:

Both notched and unnotched specimens in bending failed by nearly transverse cracks. Macroscopic examination showed no indication that fractures started on any but principal stress planes. Microscopic examination showed that some fractures might have started on shear planes.

Unnotched specimens tested in torsion at high values of stress fractured by developing longitudinal cracks at the surface spaced at intervals around the circumference. About 10 to 15 of these cracks eventually predominated and spread in depth to the center of the specimen and in length to about $\frac{3}{16}$ in. At this point the specimen had the torsional flexibility of a parallel-strand wire rope. The elements formed by the longitudinal cracks were then subjected to bending stresses with the greatest bending moments at the ends of the cracks. Hence the cracks ceased to propagate by slip and propagated by separation on planes of maximum tension stresses until the cracks intersected and the specimen fell apart.

At lower stresses in torsion, the number of longitudinal cracks was less and, at still lower stresses, a circumferential shear crack occasionally developed part way around the specimen. At the lowest stresses one or two longitudinal shear cracks started and progressed for a short distance; then the mode of propagation changed from a shear fracture to a separation type of fracture, roughly spiral-shaped.

Notched specimens tested in torsion fractured on transverse shear planes at high stresses which produced fracture in less than 31,000 cycles. The fracture planes were rather smooth and somewhat burnished by rubbing of the surfaces in the later stages of the test. At lower stresses the fractured surface had a many-pointed star appearance. Fractures had started at several nuclei around the circumference of the notch. The end view of the fracture was much the same as that of the unnotched torsion specimens. However, the longitudinal shear cracks, if they preceded the separation cracks, were not readily apparent. Instead short shear cracks on a transverse plane appeared to have been present at intervals around the specimen at the root of the crack.

At still lower stresses the number of nuclei at which fracture started was reduced, and at even lower stresses only a single nucleus was apparent. At these stresses the crack propagated diagonally across the notch out into the section of larger diameter. Evidently the stress

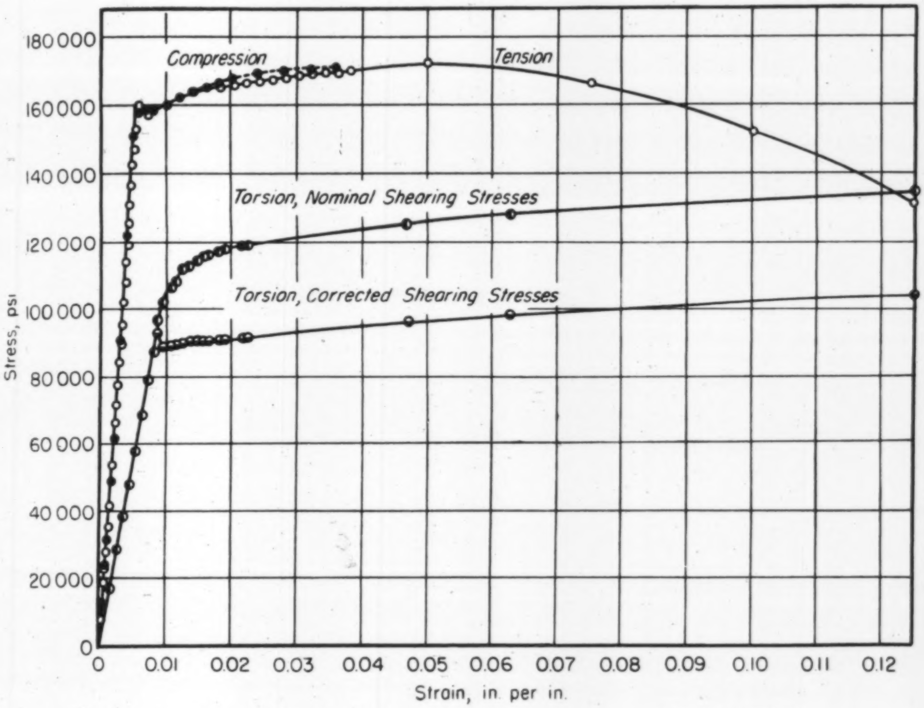

FIG. 4.—Stress-Strain Curves for Tension, Compression, and Torsion Tests.

TABLE I.—STATIC TESTS.

	Elastic Modulus, psi	Yield Point, psi		Yield Strength at 0.2 per cent Offset, psi	Ultimate Strength, psi	True Fracture Stress, psi	Reduction of Area, per cent	Elongation, per cent	Strain Rate, in. per in. per. min.
		Upper	Lower						
Tension tests, average of two tests:									
Normal stress	29.4 × 10^6	159 300	158 100		171 600	273 000[a]	55.6	13	0.0006
Shearing stress		79 600	79 000		85 800	136 500[a]			
Compression tests, average of two tests:									
Normal stress	30.3 × 10^6			158 700	172 000[b]				0.0007
Shearing stress				79 300					
Torsion test,[a] shearing stress:									
Nominal stress	11.3 × 10^6			108 000	142 500				
Stresses corrected for yielding		93 000	89 200			107 000			

[a] One test.
[b] Did not fracture.

concentration of the crack was much greater than that of the notch.

STATIC TESTS

Representative stress-strain curves for tension, compression, and torsion are shown in Fig. 4. Several static properties measured from such curves are given in Table I. The values of stress in the torsion test have been corrected as described later for the nonlinear stress distribution resulting from yielding. The corrected stress-strain diagram is also given in Fig. 4. The presence of the yield point in the corrected stress-strain curve for the torsion tests but not in the nominal stress-strain curve should be noted. The true fracture stress in tension was determined by dividing the load at fracture by the minimum area after fracture, and the true fracture stress in torsion was calculated from the torque at fracture on the assumption that the stress on the cross-section of the test specimen was constant along any diameter. This calculation yields the results that the corrected ultimate strength is equal to three-fourths the nominal value regardless of the diameter.

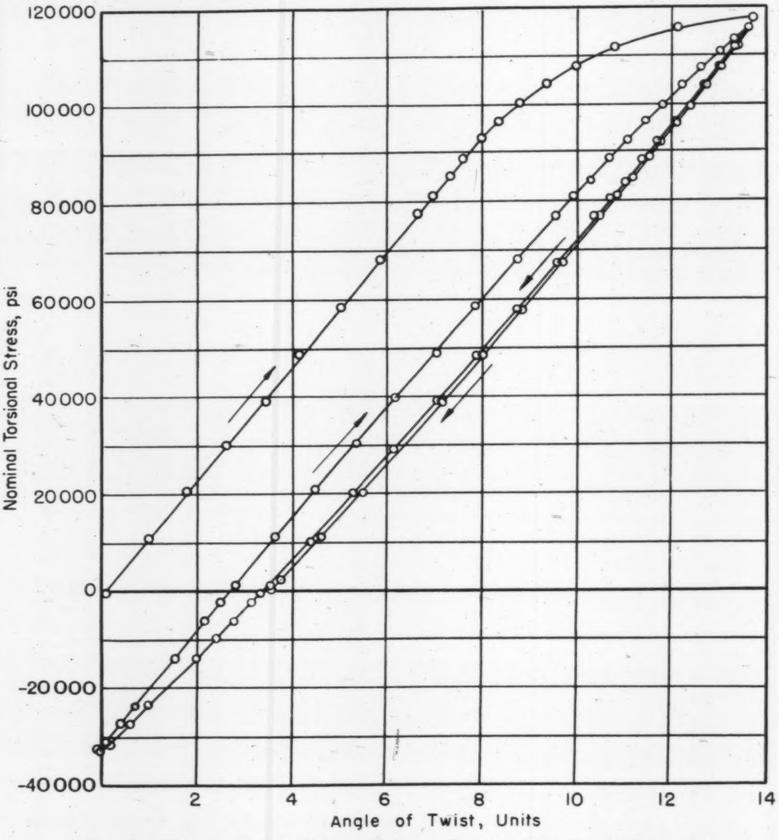

FIG. 5.—Torque-Twist Test of Fatigue Specimen in Static Torsion.

From the data in Table I, it was found

that the ratio of lower yield point in tension to the lower yield point in torsion was 1.78. Comparing this value with the values predicted by the several theories given in Table II of a previous paper (29) it was found that the energy of distortion and octahedral shear stress theories predict a ratio of $\sqrt{3} = 1.73$. The value of the ratio predicted by the total energy of deformation theory depends on Poisson's ratio. Poisson's ratio has been determined from longitudinal and transverse strains in a tension specimen to be 0.28 for the particular heat of steel tested in this program but at a Rockwell C hardness of 25. Poisson's ratio computed from the values of the moduli given in Table I is 0.32. Using the value of 0.3 the ratio of yield strength predicted by the total energy of deformation theory is only 1.61.

A static test beyond the proportional limit is illustrated in Fig. 5 for torsion of a specimen of the same shape as the fatigue specimen. The range of loads imposed was the same as that of the highest stressed specimen of the fatigue test series. Above the proportional limit a decided curvature of the diagram was observed on the first loading. On unloading, the curve was nearly linear and at the same slope as the initial loading line down to about 60,000 psi.

Below this stress a Bauschinger effect was apparent in the slight curvature to the left. On reverse loading the initial curve again had the same slope as the initial loading line and, above about 40,000 psi, it curved to the right forming a nearly closed loop.

CORRECTION OF MEAN STRESSES FOR EFFECT OF YIELDING

In this investigation the mean stress was extended to as large values as possible. This produced maximum stresses that were above the yield point of the material for some of the stresses. When this was the case, the actual maximum stresses were less than the values computed from the ordinary elastic equations. Also the alternating stresses were higher than computed from the ordinary equations as described later.

The tests at zero mean stress and at some of the small values of mean stress did not require correction since the maximum stress was below the yield point. Also, no correction was attempted for the yielding that occurred in the notched specimens at all mean stresses except zero.

Corrections for Yielding in Bending:

The corrections employed followed a semi-graphical procedure based on an extension of the Herbert equation by Morkovin and Sidebottom (30). The steps involved were as follows:

The stress-strain curve in tension was found to be nearly the same as that in compression for the range of strain employed in the fatigue tests. Hence the analysis was based on relationships applicable only when the stress-strain relation was the same in tension and compression.

From two static-tension and two static-compression tests, an average stress-strain curve was constructed and the stress and strain at the apparent proportional limit was obtained. From this curve a dimensionless stress-strain curve was constructed by dividing the stresses and strains by their corresponding value at the proportional limit. Since the plastic stress-strain diagram was slightly curved above the proportional limit, a point-by-point graphical procedure described by Sidebottom (31) was employed to construct a dimensionless moment-strain diagram.

The corrected maximum stress for any specimen could then be obtained by determining the dimensionless bending moment for the specimen and locating the corresponding value of the dimensionless strain from the moment-strain curve. The dimensionless stress corresponding to the maximum stress was then found from the dimensionless stress-strain curve at the same value of dimensionless strain. From the dimensionless stress the corrected maximum stress σ_A was calculated for each specimen.

The corrected minimum stress of the cycle was determined from the geometry of a stress-deflection diagram for loading and unloading in bending (similar to Fig. 5). It can be shown that the corrected minimum stress σ_C for the test procedure employed is given by the expression:

$$\sigma_C = \sigma_A - \sigma_F \left(\frac{\Delta_{\max}}{\Delta_{\min}} - 1 \right) \ldots\ldots (1)$$

where:

σ_A = the corrected maximum stress,

σ_F = the nominal minimum stress,

$\Delta_{\max}$ = the maximum deflection of the specimen and loading arm, and

$\Delta_{\min}$ = the minimum deflection of the specimen and loading arm.

The corrected alternating stress and mean stress were then calculated from half the difference and half the sum, respectively, of the corrected maximum and minimum stresses.

The corrected values of the alternating stresses are the values plotted in Fig. 2. The values of the corrected mean stress (where a correction was needed) are shown in thousands of pounds per square inch by the numbers adjacent to the plotted points in Fig. 2. Correction of the alternating stresses made considerable difference. Before correction the curves were spaced farther apart and the left-hand portion of the curves for high mean stresses was much flatter.

It should be noted that the corrections employed probably do not give exact values of the stresses even in the first cycle of stress because of several simplifications from actual material behavior. Among these are the following: the yielding probably was not homogeneous or continuous, and the specimen shape confined yielding to a small region which may have further complicated the geometry of plastic straining; the rate of straining in the static tests was much slower than the rate of straining during fatigue tests; and, at a few of the highest stresses, the Bauschinger effect influenced the results. In addition, the stresses change somewhat with time as discussed in a later section.

Corrections for Yielding in Torsion:

The graphical technique for correcting the stresses in torsion was that outlined by Nadai (**32**) for determining the shearing stress at the surface of a specimen of circular cross-section having a nonlinear stress-strain relation and subjected to a torque. This technique required a curve of torque *versus* angle of twist for a cylindrical specimen of circular cross section. Such a curve was obtained as shown in Fig. 4. The Nadai procedure was applied to this curve to determine the corrected shearing stress at the extreme fiber caused by the torque.

From the results of these calculations, a corrected shearing stress *versus* shearing strain curve was constructed (Fig. 4); and a curve of nominal shearing stress *versus* corrected shearing stress was constructed. By means of the latter curve the maximum stress in the cycle was determined for each specimen tested in torsion.

The corrected minimum stress, corrected alternating stress, and corrected mean stress were determined for torsion in the same manner as described for determining these values for bending. The corrected values of the alternating stresses are the values plotted in Fig. 3. The values of the corrected mean stress (where a correction was needed) are shown in thousands of pounds per square inch by the numbers adjacent to the plotted points in Fig. 3. It should be observed that the same limitations apply to this correction procedure as apply to the bending procedure.

CHANGES IN STRESS WITH TIME

In the previous sections corrections have been described which permit approximate determination of the stresses existing at the start of fatigue tests in which yielding had occurred. The question of the permanence of these stresses will now be examined.

Loss of Residual Stresses by Repeated Stressing:

The possibility exists that residual stresses in some instances may be reduced by repeated stressing. It seems doubtful, however, that residual stresses induced by the yielding resulting from the deflection cycle imposed in fatigue tests at high mean stresses (as in the present tests) would be reduced, except when stress relaxation due to creep occurred or the Bauschinger effect was encountered in the unloading portion of the cycle. If the induced residual stresses were reduced by repeated stressing (in the absence of creep) it can be shown that the maximum stress, for a constant maximum bending moment, would increase and the deflection would decrease.

This results from the following: If the residual stresses in the extreme fibers of the beam resulting from plastic bending decrease while the bending moment remains zero, static equilibrium requires the stresses in the interior fibers to decrease also. When the original bending moment is reapplied, statics requires the stress in the extreme fibers to be greater and that in the interior less than they were previously. Since the stresses in the interior are elastic, their gradient is proportional to the curvature of the beam. Since the interior stresses become less under the conditions described, the curvature and hence the deflection of the beam will be less for the same bending moment.

Alternatively, if the deflection was maintained constant, the maximum stress would be even higher and the maximum bending moment would increase. The more the residual stresses decreased, the more the bending moment would increase. Such an effect was not observed in tests similar to the present one (33). On the contrary, the bending moment was observed to decrease in some softer alloys at the beginning of the

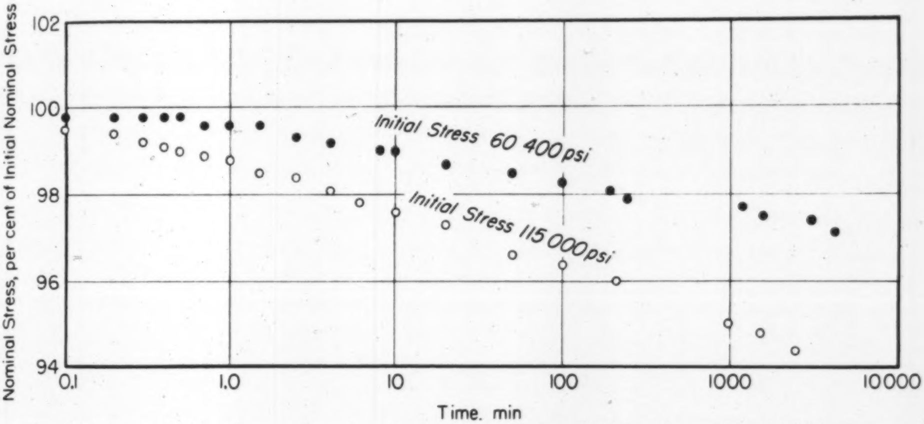

FIG. 6.—Stress Relaxation at Constant Deflection of Unnotched Specimen (Fig. 1(*a*)) in Torsion.

tests (probably due to creep) and again near the end of the test when cracks had formed (33). The latter decrease in bending moment was caused by a loss in section modulus due to the cracks.

Stress Relaxation:

Stress relaxation is encountered in steels at high temperatures and at room temperature when the sustained strain is high enough. The testing procedure employed in the present fatigue tests did not permit detection of stress relaxation during the tests. However, supplementary tests have been performed to provide an indication of the extent of stress relaxation that may have been involved. These tests were conducted on a machine of the type described in reference (33). In these tests the connecting rod of the fatigue machine was held in a fixed position and the desired initial stress was rapidly applied to the specimen by driving the mean stress adjustment with a speed wrench. The dynamometer was then read at intervals to determine the change in bending or twisting moment. This was not a precision stress-relaxation test since the strain was not kept exactly constant, but it represented the conditions of the fatigue tests rather closely, except that in the fatigue tests the crank of the fatigue machine was often turned over several times while the machine was being adjusted to the desired deflection.

The results obtained are shown in Fig. 6 for tests in torsion, one at a nominal stress representing approximately the highest nominal stress employed in the torsion fatigue tests and the other at a stress of about $\frac{2}{3}$ the upper yield point. Torsion tests were selected for this study because more material was subjected to high stress in them than in the bending tests and, therefore, they were more sensitive to relaxation.

Figure 6 shows that the nominal stress decreased about 2.3 and 5 per cent in 1000 min for initial nominal stresses of 60,400 and 115,000 psi, respectively (1000 min corresponds to about 1.7 $\times 10^6$ cycles in the fatigue tests). Figure 6 indicates that stress relaxation may cause changes in the mean or maximum stress during a fatigue test even for stresses below the yield point.

The relation between the nominal and actual stresses in the relaxation tests is not known. However, examination of studies of stress distribution in beams that creep (34, 35) suggests that the per cent decrease in the actual maximum stress in the specimen may not differ greatly from the per cent decrease in the nominal stress. That is, the shape of the stress distribution in the member may not change greatly during relaxation.

It is probable that the relaxation which occurred in these relaxation tests was substantially more than that occurring in the fatigue tests having the same maximum stress since in the fatigue tests the maximum stresses were applied only a small portion of the total time.

It should also be noted for the highest alternating stresses in fatigue tests that the duration of these tests was shorter than that of the relaxation tests so that the amount of relaxation of stress was correspondingly reduced.

In view of the above discussion, it seems possible that relaxation of stress may have caused a reduction of the mean stress (and also the maximum stress) during the fatigue tests in addition to the reduction in stress caused by yielding. The magnitude of this reduction in mean stress is probably 5 per cent or less depending on the maximum stress and duration of the test. Compared to the approximately 50 per cent or more reduction in mean stress resulting from yielding at high stresses, a correction for stress relaxation is a second-order correction and no attempt has been made to correct the present tests for this effect.

The alternating stresses in the fatigue tests probably were not affected by stress relaxation since the amplitude remained constant and the elastic modulus and linearity of the stress-strain curve on rapid unloading above the Bauschinger effect presumably remained the same.

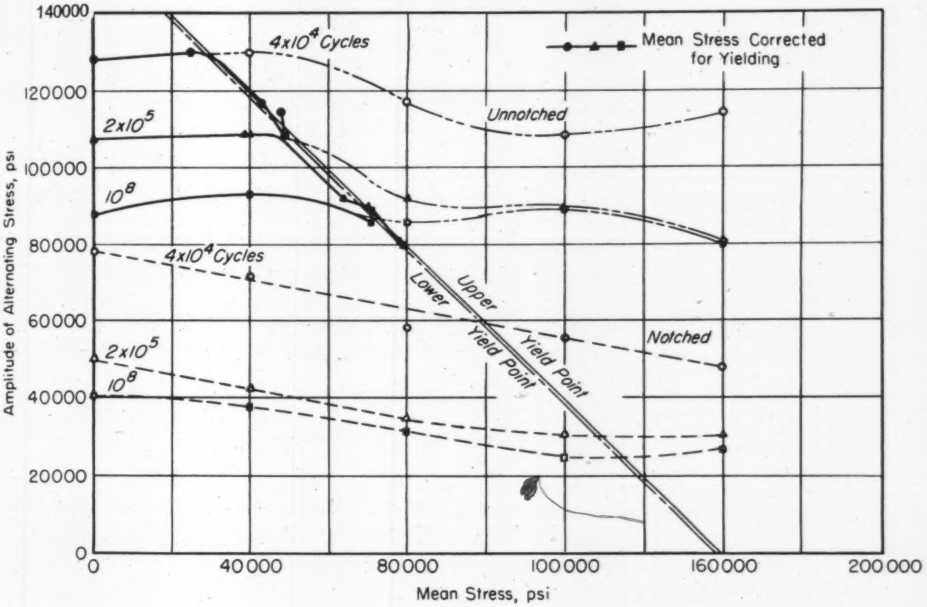

FIG. 7.—Alternating *versus* Initial Mean Stress for Bending Fatigue Tests of Notched and Unnotched Specimens.

EFFECT OF MEAN STRESS

The effect of the mean stress on the endurance limit (at 10^8 cycles) and the fatigue strengths at 4×10^4 or 2×10^4 and 2×10^5 cycles are shown in Figs. 7 and 8 for bending and torsion tests, respectively. Both nominal and corrected mean stresses are shown. The alternating stresses shown (corrected as described above) represent ordinates to the *S-N* curves at the given number of cycles, and the corrected mean stresses (shown as blacked-in test points) were obtained by interpolating to find the mean stress corresponding to the ordinate to the *S-N* diagram in question.

Perhaps the most important observation from these tests is that increasing the mean stress caused only a small change in the fatigue strength or endurance limit for either torsion or bending, especially within the elastic range. As might be expected, correction of the mean stresses for yielding reduced the mean stress approximately to the lower yield point line (see Figs. 7 and 8).

In bending, the yielding appeared to reduce the fatigue strength slightly. In torsion both an increase and a decrease were observed.

These results tend to support the suggestion made earlier (**24, 25**) by one of the authors that the fatigue strength of a material is associated with the alternating stress and that the major effect of a mean stress is to alter the effective structure of the material. If the effective structure of a material is only slightly altered by yielding or elastic deformation, only a slight change in the fatigue strength with mean stress would be observed. If this suggestion is correct, then the pronounced effect of mean stress observed for some materials would indicate that the mean stress produced structural changes in the material to which the fatigue strength was sensitive.

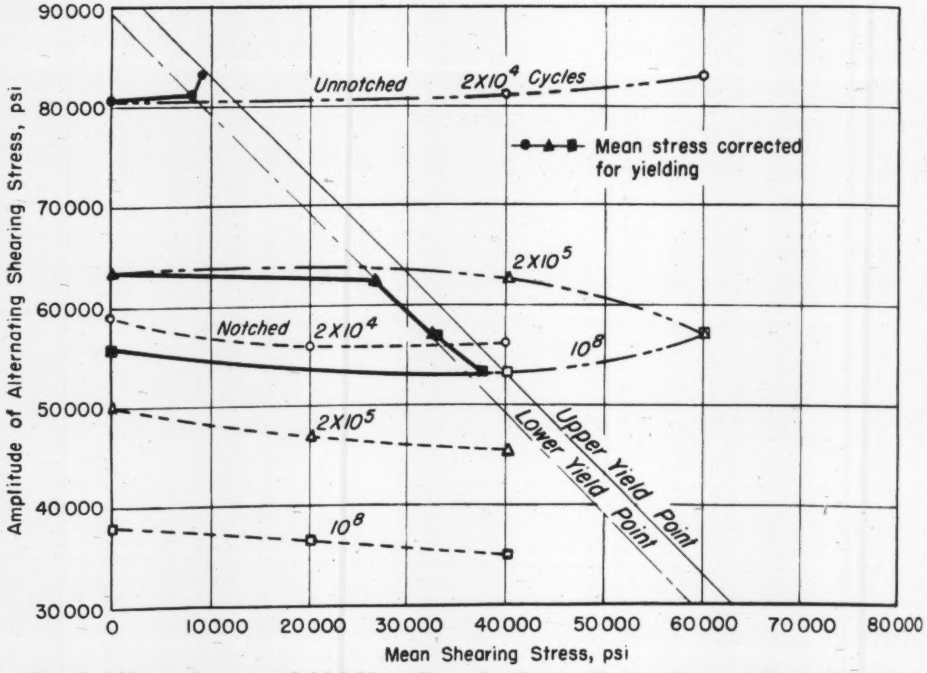

FIG. 8.—Alternating *versus* Initial Mean Stress for Torsion Fatigue Tests of Notched and Unnotched Specimens.

The fact that the fatigue strength of notched specimens in both bending and torsion decreased with increasing mean stress may be explained by the fact that the actual maximum stresses produced in the notched specimens caused yielding in all except the tests at zero mean stress. The fatigue notch factor (36) for the circumferential notch shown in Fig. 1(*b*) at zero mean stress in bending was 1.64, 2.14, and 2.17 at 4 × 10^4, 2 × 10^5, and 10^8 cycles, respectively. For torsion of a specimen containing the same circumferential notch, the fatigue notch factor at zero mean stress was 1.36, 1.27, and 1.47 at 2 × 10^4, 2 × 10^5 and 10^8 cycles, respectively. It is evident that application of either these factors or the theoretical stress concentration factors (2.6 for bending, 1.65 for torsion) to the maximum stress results in nominal stresses which exceed the yield point for all specimens except those at zero mean stress and those in the vicinity of approach the endurance limit for the lowest mean stress.

In view of the above, it is difficult to see how one can compute a fatigue notch factor that is meaningful at a mean stress different from zero unless the actual mean stress in the notched specimen is known to equal that in the unnotched specimen.

ALTERNATING *VERSUS* MAXIMUM STRESS

If, as seems to be the case, general yielding affects the fatigue strength, then perhaps mean stress is not the significant parameter. Perhaps the maximum stress would be a more logical parameter. The fatigue strengths at three different numbers of cycles have been plotted as a function of the maximum stress in the cycle in Fig. 9 for unnotched specimens in bending and torsion.

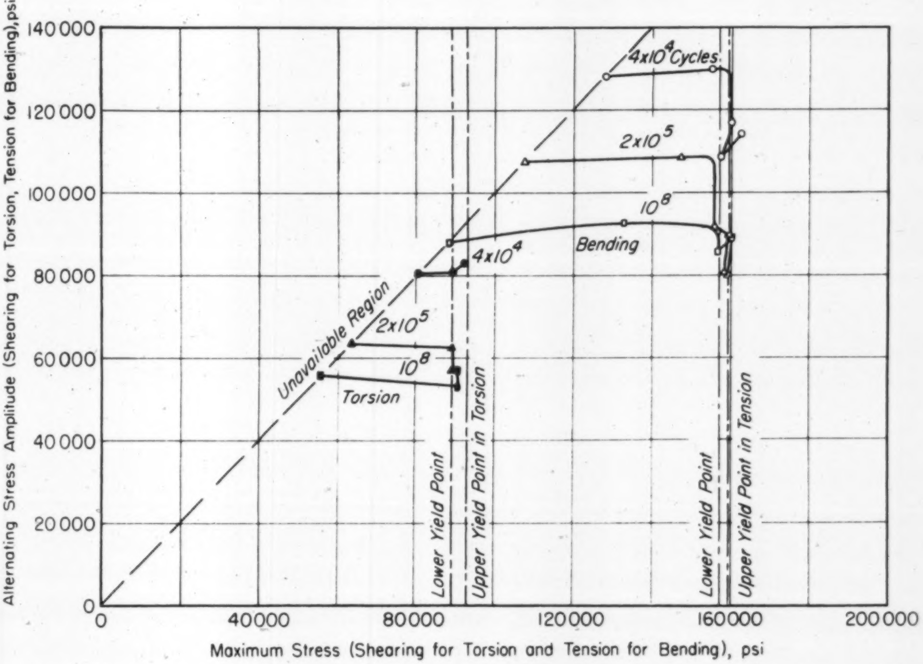

FIG. 9.—Alternating *versus* Initial Maximum Stresses for Bending and Torsion Fatigue of Unnotched Specimens. Stresses shown are corrected for yielding.

In this diagram the area above the diagonal line is unavailable for testing in tests of the type performed. For axial loading this region would be available and, with specimens of unsymmetrical cross-section, this region can be made available for bending.

In view of the above comments, an often referred to diagram[6] of the type shown in Fig. 9 may be misleading in that it implies that all parts of the diagram are available for torsion testing. The possibility of misinterpretation is increased by the fact that data from many different materials are plotted on one diagram in such a way that the zero-mean-stress point from each material is plotted at different abscissas but the same ordinate, thus giving more weight to the horizontal character of the curve then is justified.

It is evident from Fig. 9 that an increase in maximum stress causes no significant change in the alternating stress (fatigue strength) until yielding occurs, at which time the fatigue strength generally decreased and then in some instances increased again.

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[6] Figure 4 of paper by Smith (**1**), Maximum Stress Alternating Stress Diagram for Fatigue Tests of Notch-Free Cylindrical Torison Specimens of Twenty-seven Ductile Metals.

While the maximum stress seems to be the most logical parameter from a theoretical standpoint, it is possible that from a design standpoint the mean stress may be more useful in describing the range of stress because of the unavailable region in such diagrams for certain cases.

FATIGUE NOTCH FACTOR AND STRESS CONCENTRATION FACTOR

It has become accepted practice in many places, when designing a part containing notches and subjected to fluctuating loads, to apply a fatigue notch factor to the alternating component of the nominal stress but not to the mean stress. The above discussion suggests that the reason this is good design practice may result from the possibility that the effects of alternating stresses in producing fatigue fracture and the effect of maximum stresses (and hence mean stresses) are independent except through the medium of changes in the structure of the material caused by the maximum stress.

Perhaps a better way of considering the problem would be to apply the theoretical stress concentration factor to the maximum stress to determine whether yielding occurred and what maximum stress existed. Then a fatigue notch factor would be applied to the alternating stress. This result would be compared with an *S-N* diagram of the given material as modified by the structural change expected from the calculated maximum stresses. The modification of the *S-N* diagram (and hence fatigue strengths, endurance limit, etc.) resulting from structural changes due to yielding is probably different for different materials.

EFFECT OF STATE OF STRESS

The ratio of the fatigue strength in bending, b, to that in torsion, t, was found to be 1.69 at 2×10^5 cycles and 1.57 at the endurance limit. Referrring again to Table II of a previous paper (**29**), it is found that the theory of failure which comes closest to predicing these values is the total energy of deformation for which the value of b/t is 1.61 when a value of Poisson's ratio of 0.3 is used. The theories which next most nearly predict the observed values of b/t are the energy of distortion theory and the octahedral shear stress theory, both of which predict a value of $\sqrt{3}$.

The values of b/t obtained from the ratio of the nominal stresses in notched specimens yield the surprising result of 1.0 at 2×10^5 cycles and 1.06 at the endurance limit. However, if the nominal stresses are corrected in accordance with the theoretical stress concentration factors K_{tb} for bending and K_{tt} for torsion, the following relation is obtained:

$$\frac{b}{t} = \frac{K_{\text{tb}}\sigma_{\text{nominal}}}{K_{\text{tt}}\tau_{\text{nominal}}} \qquad (2)$$

From this relation, values of b/t of 1.58 at 2×10^5 cycles and 1.68 at the endurance limit are found. These values are nearly the same as those determined from the unnotched specimens but are reversed in order.

Examination of the literature discloses that the values of b/t for unnotched cast iron specimens range from about 0.9 to 1.2 which is in the range of the value of b/t obtained from the notched specimens of SAE 4340 steel. Since cast iron is known to contain many notches caused by graphite-flake inclusions, perhaps the low value of b/t obtained for cast irons results from the operation of different stress concentration factors for the internal notches in bending and in torsion. The fact that the graphite flakes are considered to be randomly oriented, however, tends to discount the plausibility of this theory.

The degree of anisotropy in the

fatigue properties of the steel tested in this program is not yet known. However, tests are under way to obtain this information for the same steel heat treated to a Rockwell C hardness of 25. It is possible that anisotropy may have affected the value of b/t as discussed previously (**24, 25**).

CONCLUSIONS AND OBSERVATIONS

1. An increase in the mean stress in either bending or torsion of unnotched specimens caused only a small change in the fatigue strength (defined in terms of the alternating stress) until yielding occurred, at which stress the strength generally decreased.
2. A slight decrease in the fatigue strength of notched specimens was observed in both bending and torsion when the mean stress was increased. However, most mean stresses employed above zero produced yielding.
3. The process of fatigue fracturing in bending and torsion was described and the influence of stress on the mode of crack formation observed.
4. The mean and alternating stress values were corrected for the nonlinear stress distribution resulting from yielding.
5. Possible changes in stress due to loss of residual stress and relaxation of stress have been examined.
6. The corrected values of the lower yield point in torsion and lower yield point in tension agreed best with the relation predicted by either the energy of distortion theory or octahedral shear stress theory.
7. The fatigue strengths in bending and torsion agreed best with the relation predicted by the total energy of deformation theory.
8. It is suggested that the maximum stress in a cycle may be a more significant parameter than mean stress.
9. The fatigue notch factors at the endurance limit and zero mean stress were 2.17 in bending and 1.47 in torsion compared to stress concentration factors of 2.6 in bending and 1.65 in torsion.
10. It has been suggested that the fatigue strength is not influenced by mean stress or maximum stress but may be influenced by the structural changes produced by the maximum stress.

Acknowledgment:

The project was conducted in the Department of Theoretical and Applied Mechanics of the University of Illinois. The authors are grateful to the University of Illinois Engineering Experiment Station and Hamilton Standard Division of United Aircraft Corp. for financial assistance. The latter also furnished the material and machining of half of the specimens.

The authors are greatly indebted to D. G. Richards of Hamilton Standard Division for initiating this project and contributing time, effort, and ideas to it. The help of the following assistants is also gratefully acknowledged: C. P. Hendrickson, D. D. Strohbeck, D. E. Martin, A. Mahak, and C. M. Peterson.

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DISCUSSION

MR. JOSEPH MARIN.[1]—I would like to ask a question regarding your interpretation of the torsion fatigue strength data. You indicated that the distortion energy theory gives a good prediction of the torsion fatigue test results. However, some torsion fatigue test results, such as those recently published by Gough, do not agree with values predicted by the distortion energy theory. Is this discrepancy due to the fact that the test results by Gough were not corrected for the influence of plastic flow on the stress values?

MR. B. J. LAZAN.[2]—There are of course two factors which may cause a change in stress during fatigue testing under nonuniform stress, such as bending or torsion. One is the redistribution of the stress on a cross-section due to plastic action; the other is the change in effective modulus of elasticity which associates the alternating strain with the alternating stress. I would appreciate Mr. Findley's comments on the significance in his test of each of these causes for deviation between calculated and true stress.

MR. J. M. LESSELLS.[3]—These results of Mr. Findley are very interesting, but the effect of range of stress on the fatigue strength is still obscure. While the theory of fatigue, as proposed by Orowan,[4] appears to indicate that the mean stress of the cycle has no effect on the fatigue strength, the results of many materials published by Gough[5] indicates that the range decreases as the mean stress is increased. Moreover, the empirical relations suggested by Goodman and

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Gerber, and widely used by engineers, do assume such a behavior.

Regarding the remarks by Mr. Marin on the work of Gough[6] on combined stresses, very good agreement was obtained for the cases of a low carbon steel and an alloy steel with the Mises-Hencky theory. These results have also been summarized by Peterson.[7]

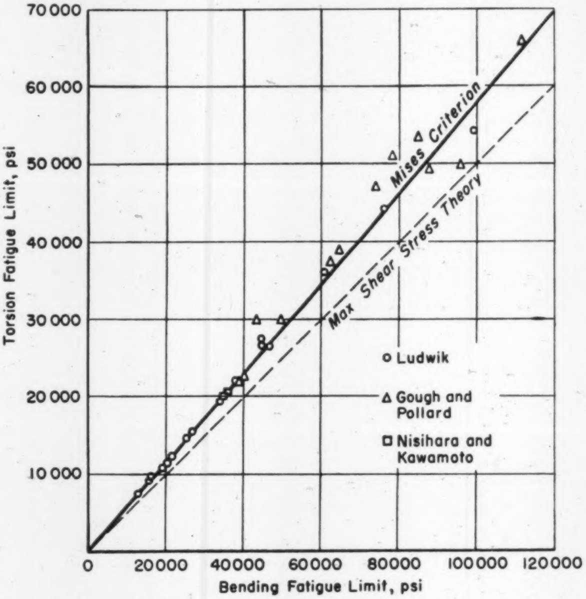

FIG. 10.—Comparison of Torsion and Bending Fatigue Limits for Ductile Materials.

MR. R. E. PETERSON[8] (*by letter*).—One of the conclusions indicates agreement with "either the energy of distortion theory or octahedral shear stress theory." These theories have the same mathematical expression as the Mises criterion which was proposed[9] as a mathematical interpretation avoiding the discontinuities of the maximum shear diagram for biaxial stresses. It has been shown by Sachs[10] and by Cox and Sopwith[11] that one obtains the same yield ratio of torsion to tension as for the Mises criterion if one calculates total yielding of an aggregate of randomly orientated grains on the basis of resolved shear stress. For this reason it has been suggested by Beeching[12] that energy designations be abandoned in favor of a simpler term, such as Mises criterion.

It is rather interesting that if one considers fatigue ratios of torsion to tension (Fig. 10), rather good agreement, on the average, is also obtained with the Mises criterion. In the absence of statistical procedures it is not likely that a meaningful decision can be reached regarding theories from a single comparison.

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Since notches represent a biaxial case at the surface, one should logically utilize a strength theory in comparing theory and test. If the Mises criterion, or a similar theory, is used, this has the effect of bringing the theoretical and test values closer together. Much more data are needed to settle this matter. The authors' views would be appreciated with regard to application of strength theory in the case of notched members.

MR. W. N. FINDLEY (*author's closure*).—The discussors have raised some very important and difficult questions. In response to Mr. Marin's question, the present fatigue data for bending and torsion are nearest to the predictions of the total energy of deformation theory. The ratio *b/t* at the endurance limit was 1.57 compared to 1.61 for this theory. The energy-of-distortion theory predicts 1.73.

In the recent paper by Gough (**13**),[13] the values of *b/t* for twelve steels ranged from 1.46 to 1.93 with five values within less than 3 per cent of *b/t* for the energy-of-distortion theory. In my opinion these differences in the ratio *b/t* are not due to the fact that corrections were not made for the influence of plastic flow on the stress values. I have this opinion because in the range of the endurance limit, at least, the stresses would be elastic for tests at zero mean stress. At higher mean stresses the stresses were probably linear on unloading and reloading so that the computed alternating stresses were probably correct. Only the mean stresses at the highest values were affected by plastic flow.

Mr. Lazan asked for comments on the significance of two causes for deviation between calculated stress and true stresses. The causes of deviation between nominal stresses and actual stresses that were discussed in the paper were (*a*) yielding, (*b*) Bauschinger effect, (*c*) stress relaxation, and (*d*) crack formation. Of these (*a*) and (*c*) caused no deviation from the nominal alternating stresses, (*b*) caused a substantial deviation from the nominal alternating stresses when the amplitudes were very large, and (*d*) caused unknown but probably large deviations in both alternating and mean stresses during the latter part of the test. The mean stresses were substantially affected by (*a*) and to a lesser extent by (*c*). No change in modulus was observed in these or similar tests. An effective modulus may be associated with yielding, Bauschinger effect or hysteresis damping. The effect of damping in the absence of the other two was not determined but was probably very small.

Mr. Lessells' remarks are appreciated.

In response to Mr. Peterson's discussion I have the following comments. It is true that except for a radical sign the three theories—energy of distortion, octahedral shear stress, and Mises criterion—have the same mathematical formulation and hence produce the same results as far as a designer is concerned. However, in attempting to determine the basic laws governing fatigue failure under combined stresses, I feel that they are three distinctly different concepts of what controls fatigue failure—the first a strain energy, the second a shearing stress, and the third empiricism.

There is also a difference in the accuracy with which a given set of data agree with the first two of these theories. This comes about because the energy theory is the square of the stress theory; hence the effect of deviations from the theory is more pronounced in the energy theory. This fact, of course, does not show up if the comparison is made on the basis of stress in both cases. However, in my opinion the comparison should be made in terms of energy for an energy theory and stress for a stress theory as illustrated in earlier papers by the author (**24, 25**), for example.

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[13] The boldface numbers in parentheses refer to the list of references appended to the paper, see p. 784.

I do not wish to imply by the text or conclusions 6 and 7 that I feel the energy theories or octahedral shear stress theory describe the fundamental relationships which control fatigue under combined stress. I feel that it is unlikely that either is the correct explanation. My reasons are given in earlier papers (**24, 25**).

I understand the references given by Mr. Peterson describe theories of failure by general yielding. According to available evidence fatigue failure starts in a very localized manner on particular planes in particular crystals and fatigue is a fracturing rather than yielding process. Thus it seems that the same theory would not necessarily describe both processes.

Since the origin of a fatigue crack may involve one plane or set of parallel planes in a single crystalite it seems unnecessary that the maximum resolved shear stress in every crystalite in a given region reach a critical value before cracks start as proposed in the octahedral shear stress concept for the initiation of yieldings.

While the weight of evidence given in Fig. 10 of the discussion strongly favors the Mises criterion, another grouping of these and other data show that several rather distinct classes of materials may be formed which would show several lines like that of Fig. 10 but at different slopes, indicating apparent agreement with different theories. This material is presented in a technical report which is in preparation on Contract No. DA-11-022-ORD-995 with the Office of Ordnance Research.

It appears possible that the apparent agreement of different materials with different theories may be due to the presence in different degrees of some influencing factor such as anisotropy (**24, 25, 29**), or different rates of crack propagation by two different mechanisms (**24**).

It may also be that fatigue failure results from fluctuating shearing stress (or strain) but that the strength is influenced by (*a*) the structure of the material, which may be altered by yielding, (*b*) the presence of normal stress on the plane of the maximum shear stress, (*c*) the presence of normal stresses on the plane normal to the maximum shear planes, and (*d*) other factors. The effect of the normal stresses, like the effect of yielding, may be different for different materials. That is, normal stresses on the planes of maximum shear stress may tend to inhibit slip, if compression, or make slip easier, if tension; and the effectiveness of these normal stresses may depend on the nature of the atoms involved in the potential slip planes, presence of foreign atoms, type of lattice, etc. Some experiments to determine the effect of normal stresses on the shear stress to produce slip in single crystals, with and without imperfections such as foreign atoms, might be very enlightening.

Mr. Peterson raised a question about the application of strength theory to notched members. I feel that a satisfactory complete theory for fatigue must explain the behavior of notched members. The objections given above to the Mises criterion would, I believe, be as applicable in the case of notched members. In the data of the present paper application of the stress concentration factors for bending and torsion to the data for the notched specimens resulted in nearly the same ratio of fatigue strength in bending to that in torsion as for unnotched specimens. This may be an indication that the same theories operate in both instances.

INFLUENCE OF GRAIN SIZE ON FATIGUE NOTCH-SENSITIVITY*

By R. W. Karry[1] and T. J. Dolan[2]

Synopsis

A study was made of the reduction in flexural fatigue strength caused by a circumferential notch in specimens of a single-phase metal. Groups of specimens were prepared from 70-30 brass having the same chemical analysis but processed to obtain three different degrees of cold reduction and three different annealed grain sizes. Two notch shapes having the same depth but different root radii were investigated. It was found that the fatigue strength reduction factor and notch-sensitivity were not unique material constants; these were dependent upon both the grain size of the metal and the notch root radius. An increase in grain size produced a significant lowering of the fatigue strength, but also resulted in a decrease of notch-sensitivity and fatigue strength reduction factor. Experimentally determined fatigue strength reduction factors were somewhat smaller than those computed from H. Neuber's theory of the effect of an elementary particle on notched material behavior. On comparing fatigue strengths developed, a correlation was found between the calculated average stress on a grain located in the critically stressed surface of a notched specimen and that in an unnotched specimen having the same grain size.

One of the most important problems encountered in the design of machine parts is the prediction of the strength of a member when subjected to the stress concentrating effects developed by a geometrical notch. Several experimental investigations (**4**, **5**, **12**)[3] have disclosed that the fatigue limit or fatigue strength of notched fatigue specimens is usually greater than that predicted by use of theoretical stress concentration factors based on assumptions of elasticity. Several expressions have been developed in attempts to explain this discrepancy between classical theory and experimental results. Among these expressions, the two which have come to be commonly accepted[4] are the "fatigue strength reduction factor," K_f, and the "notch-sensitivity," q. Notch-sensitivity supposedly varies between zero, when $K_f = 1$, and unity, when $K_f = K_t$; however, values of q less than zero (negative) and greater than one have been observed in tests of several different materials. Neither K_f nor q have any significance unless the stress range, geometry, size, and material of the specimen are stated.

For conditions of *static* loading, Neuber (**3**) has discussed the failure of the classical theory of elasticity to predict adequately the behavior of metals with

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 801.

[4] K_f is defined as the ratio of the fatigue strength of specimens with no stress concentration to the fatigue strength of members with a stress raiser. Notch sensitivity, q, is a measure of the degree of agreement between K_f and K_t, where K_t is the theoretical stress concentration factor. The value of q is defined (**10**) as follows:

$$q = \frac{K_f - 1}{K_t - 1}$$

notches of very small radii. The theory of elasticity assumes the material to be perfectly homogeneous and made up of infinitesimally small particles which is *not* the case for engineering materials. If the radius of curvature of the surface is everywhere large enough, compared with the size of the crystalline structure of the material, no pronounced error in the classical theory becomes evident. To take into consideration, mathematically, the crystalline structure of metals, Neuber assumed the material to be composed of numerous small but finite structural units which he named the "elementary particle." The values of theoretical stresses in the region of peak stress were averaged over the surface of the elementary particle which reduced the value of the effective maximum elastic stress because of the stress gradient existing over the particle. Consequently, Neuber found that the stress concentration factor also depends upon the size of the elementary particle when the surface curvature is relatively sharp. It appears possible that this more realistic approach to the stress concentrating effect of a sharp notch in a polycrystalline metal may also be applicable to the case of repeated loading.

The work of Peterson (**1, 2**) lends some support to this suggestion, at least in a qualitative manner. Briefly, Peterson found that fine-grained, heat-treated steels were more notch sensitive in fatigue than coarse-grained, annealed steels when tested under similar conditions. Results obtained by other investigators (**8, 11**) also seem to indicate a possible relationship between metallurgical structure and fatigue notch sensitivity. One might reason that a stress raiser introduces a steep stress gradient at the specimen surface, resulting in an appreciable stress drop across any one grain assumed to be located at the root of the notch. For metal having large grains, there would be a relatively large stress drop and, hence, a lower average stress acting on the grain. Thus for the same ***peak*** stress calculated at the surface, the large grains are subjected to a less severe loading condition. Therefore the fatigue behavior of a notched specimen could not be expected to be the same as that of an unnotched specimen where the drop in stress across one grain is relatively small.

Although the contributions discussed above are of considerable importance, some confusion exists concerning what measure of grain size was utilized (**1, 2**) or if the elementary particle (**3**) is related to the size of a metal space lattice or to some kind of crystal grain.

A survey of the literature on fatigue notch sensitivity did not disclose any instances in which a systematic investigation of the influence of grain size was made. In view of the lack of available data on this subject, it seemed desirable that a laboratory study be made to furnish the needed information.

PURPOSE AND SCOPE

The purpose of this investigation was to study the influence of measured grain sizes on the fatigue notch sensitivity of a metal. In order to keep the number of unknown variables to a minimum, it was thought advisable to select for test a simple single-phase metal in which the grain size could readily be controlled, for this reason, a 70-30 brass was used in the experimental program. The effects of three different degrees of cold work and three different annealed grain sizes on the notch sensitivity of the 70-30 brass were determined experimentally. Notched fatigue specimens were tested at approximately 3500 rpm in rotating cantilever-beam machines under constant load at zero mean stress. Two different notch shapes were investigated to reveal the effect of notch root radius on the notch-sensitivity of the brass.

In addition, Neuber's theory of the effect of an elementary particle on notched material behavior was investigated for its applicability to the case of repeated loading.

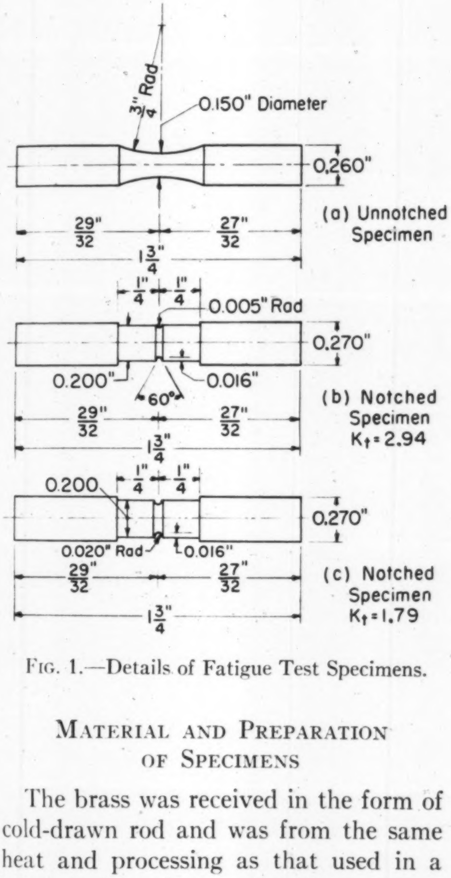

FIG. 1.—Details of Fatigue Test Specimens.

MATERIAL AND PREPARATION OF SPECIMENS

The brass was received in the form of cold-drawn rod and was from the same heat and processing as that used in a previous investigation (**13**). It was composed of 69.5 per cent copper, less than 0.05 per cent lead, 0.01 per cent iron, and the balance zinc. The various specimen groups were subjected to different mechanical treatments by the manufacturer as listed in Table I.

Notched fatigue specimens, Figs. 1(*b*) and (*c*), were first machined from groups of the "as-received" material and some of these groups were later given specific annealing treatments to alter the grain size. The theoretical stress concentration factors, K_t, were determined by means of Neuber's nomograph (**3**). The unnotched fatigue specimen, Fig. 1(*a*) was used by Sinclair and Craig (**13**); it is included in this paper since the unnotched fatigue strengths previously determined by Sinclair and Craig for specimens from the same groups of bar stock were utilized in analyzing the results.

TABLE I.—"EQUIVALENT" GRAIN SIZES OF COLD-DRAWN BRASS.

Group Designation	Ready-to-Finish Grain Size, mm	Cold Reduction in Area, per cent	Final Diameter of Rod, in.	Equivalent Grain Size, mm
A	0.015	20	0.324	0.00295
D	0.015	40	0.281	0.00205
G	0.015	60	0.229	0.00185

In the case of cold-worked metals, it is practically impossible to determine the grain size of the material by using optical methods. However by introducing a new concept, Sinclair and Craig (**13**) determined an "equivalent" grain size for cold-worked brass. They determined the mean grain diameters, by Jeffries' planimetric method,[5] and Rockwell hardness numbers of specimens subjected to various annealing treatments. These values are plotted as solid black dots in Fig. 2. It had previously been shown (**7**) that a straight-line relation appears to exist between the log of the mean grain diameter and the Rockwell hardness of annealed brass. Sinclair and Craig reasoned that if the Rockwell hardness number of a cold-worked brass is known, an "equivalent" grain diameter

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[5] Tentative Methods for Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79 – 49 T), 1952 Book of ASTM Standards, Part 2, p. 1146.

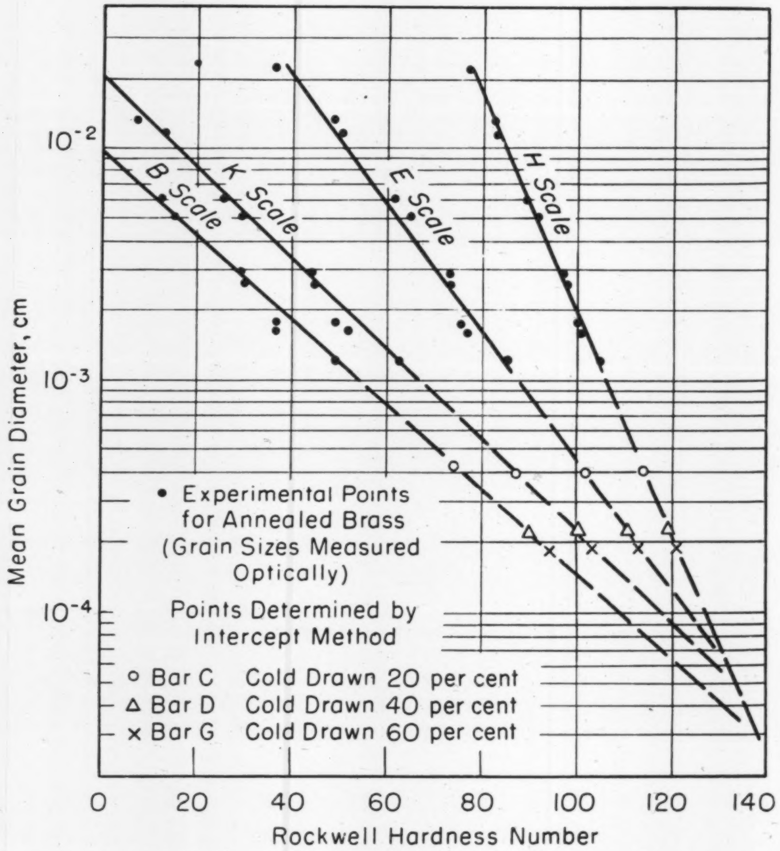


FIG. 2.—Effect of Mean Grain Diameter on Rockwell Hardness Number.
Each point is an average value for at least ten hardness readings. From Sinclair and Craig (**13**).

TABLE II.—ANNEALING TREATMENTS AND GRAIN SIZES OF SPECIMENS.

Stress Concentration Factor, K_t	Group Designation	Anneal Temperature, deg Fahr	Time at Temperature, hr	Approximate Annealed Grain Size, mm
2.94	G	750	4	0.0125
	A	960	4	0.033
	A	1200	4	0.125
1.79	A	960	4	0.023
	A	1200	4	0.107
	A	1400	2	0.28

TABLE III.—UNNOTCHED FATIGUE STRENGTHS AT 100 MILLION CYCLES.[a]

Brass Group	"Equivalent" or Mean Grain Size, mm	Fatigue Strength at 10^8 Cycles, psi
G	0.00185	40 000
D	0.00205	30 000
A	0.00295	24 000
Anneal G	0.012	22 000
Anneal A	0.026	17 000
Anneal A	0.131	12 000
Anneal A	0.305	9 000

[a] From Sinclair and Craig (**13**).

may be established by the intercept method on an extrapolation of the curves in Fig. 2. These estimated grain sizes were observed to be "equivalent" as far as their general effects on the mechanical properties of the brass were concerned. The term grain size as used throughout the remainder of this paper will refer to the "equivalent" grain sizes of cold-worked brass and the mean grain sizes (Jeffries' method) for annealed brass. The grain sizes of the as-received groups studied are given in Table I.

TABLE IV.—NOTCHED FATIGUE STRENGTHS AT 100 MILLION CYCLES.

Stress Concentration Factor, K_t	"Equivalent" or Mean Grain Size, mm	Fatigue Strength at 10^8 Cycles, psi
2.94	0.0018	13 000
	0.0020	12 000
	0.0029	12 000
	0.012	12 000
	0.033	10 000
	0.125	9 000
1.79	0.0018	23 000
	0.0020	21 000
	0.0029	16 000
	0.023	12 000
	0.107	10 000
	0.28	9 000

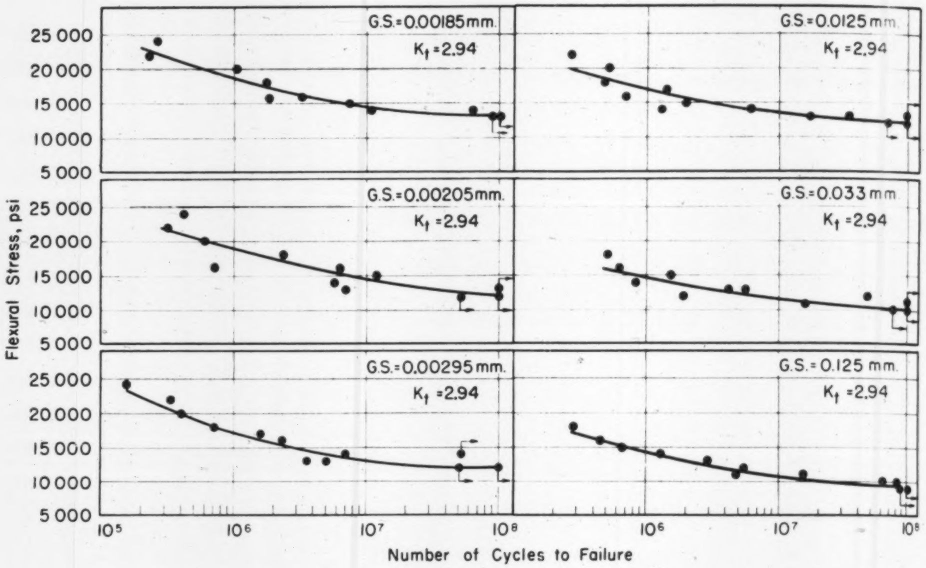
After machining, a number of notched specimens were given various annealing treatments as listed in Table II. The annealed grain sizes resulting from these treatments were also determined from the hardness-grain size relation of Fig. 2. The annealed grain sizes for specimens of each notch are given in Tables II to IV. The differences in grain size for the same annealing treatment resulted from heating the batches of specimens of each notch separately. The Rockwell hardness number of each group given an annealing treatment was the average value of at least 20 hardness readings. These hardness readings were obtained on specimens which were approximately $1\frac{3}{4}$ in. in length and which had flats milled on two sides of the rod. The hardness readings for the various groups agreed closely with the data plotted in Fig. 2 for the different grain sizes investigated.

The test section of each specimen was polished electrolytically, using a solution of 530 g per l of orthophosphoric acid in water, at a temperature of 100 ± 1 F for a period of 10 min as the final step preparatory to testing. The electropolishing removed approximately 0.001 in. of metal from the diameter, uniformly along the length of the test section.

TABLE V.—INFLUENCE OF "EQUIVALENT" OR MEAN GRAIN SIZE ON THE FATIGUE STRENGTH REDUCTION FACTOR AT VARIOUS NUMBERS OF CYCLES.

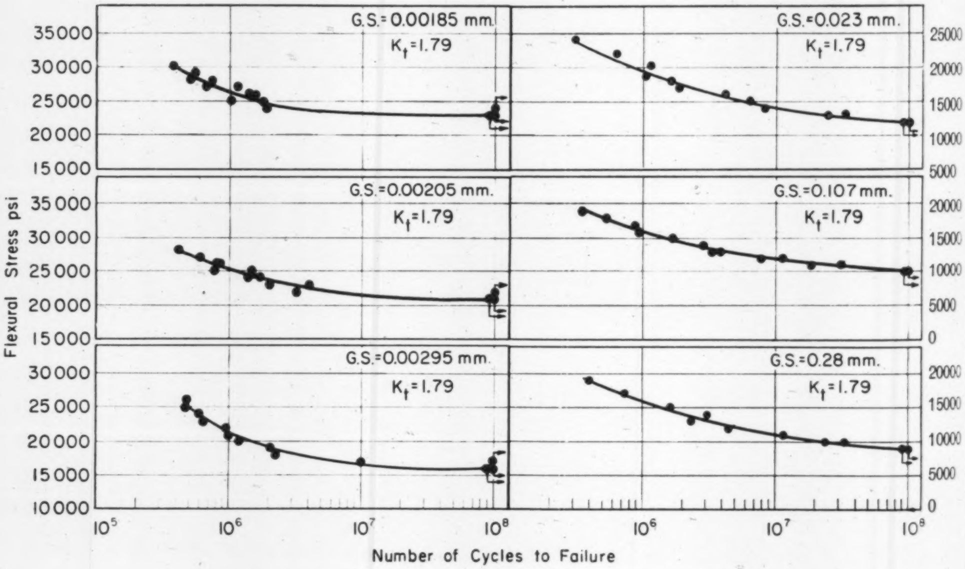
Stress Concentration Factor, K_t	"Equivalent" or Mean Grain Size, mm	Fatigue Strength Reduction Factor, K_f					
		Number of Cycles					
		5×10^5	10^6	5×10^6	10^7	5×10^7	10^8
2.94	0.00185		2.86	2.76	2.84	3.01	3.10
	0.00205	2.54	2.46	2.19	2.19	2.29	2.46
	0.00295	2.01	1.97	1.85	1.92	2.06	2.12
	0.0125	1.85	1.83	1.72	1.73	1.89	1.82
	0.033	1.75	1.72	1.63	1.59	1.66	1.60
	0.125	1.61	1.54	1.46	1.37	1.27	1.33
1.79	0.00185		2.12	1.84	1.79	1.74	1.70
	0.00205	1.88	1.84	1.55	1.51	1.47	1.56
	0.00295	1.55	1.58	1.48	1.53	1.52	1.57
	0.023	1.42	1.44	1.42	1.42	1.44	1.46
	0.107	1.34	1.34	1.31	1.28	1.20	1.28
	0.28						1.04

After polishing, all specimens were measured on three different diameters, and the notch contour checked at 50× on an optical microprojector. For any one specimen the maximum difference in diameters was observed to be less than 0.001 in. For a majority of the specimens this difference was observed to be less than 0.0005 in. The average value of the specimen diameters at the root of the notch was used to calculate the nominal stress in the specimen by the elementary flexure formula, $S = \frac{Mc}{I}$.



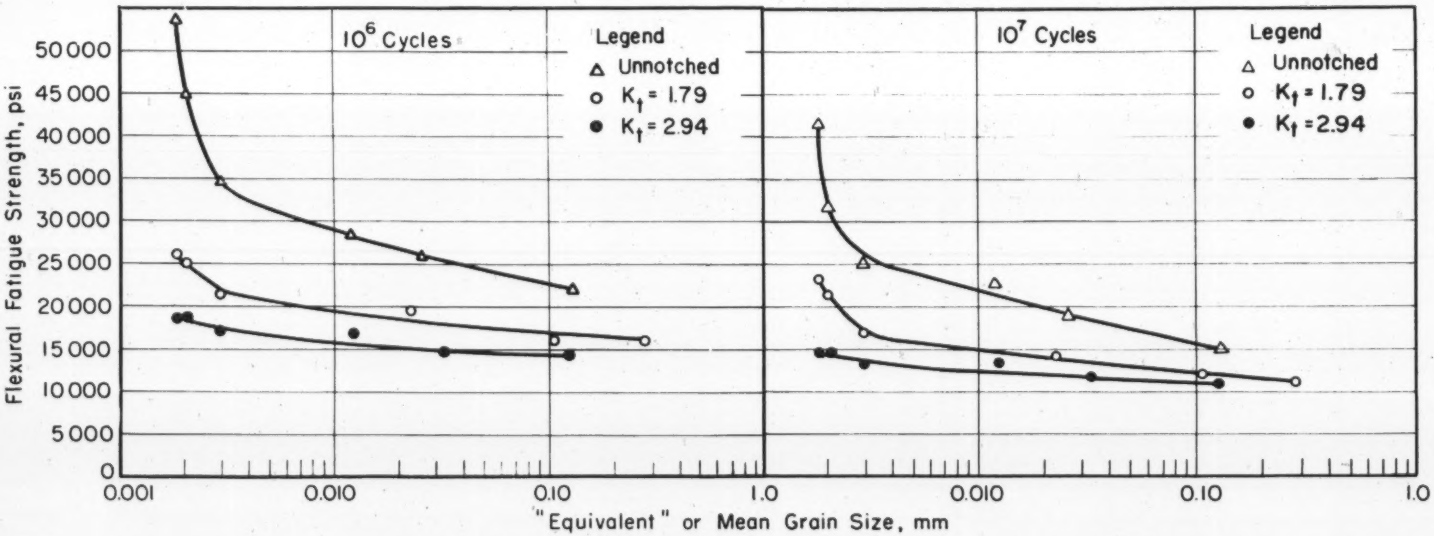
(*a*) "Equivalent" grain size. (*b*) Annealed grain size.

FIG. 3.—Influence of "Equivalent" and Annealed Grain Size on Fatigue Strength of 70-30 Brass.



(*a*) "Equivalent" grain size. (*b*) Annealed grain size.

FIG. 4.—Influence of "Equivalent" and Annealed Grain Size on Fatigue Strength of 70-30 Brass.



(*a*) At 10^6 cycles.

(*b*) At 10^7 cycles.

FIG. 5.—Influence of "Equivalent" or Mean Grain Size on the Fatigue Strength.

RESULTS OF TESTS AND DISCUSSION

The fatigue strengths at 100 million cycles for the unnotched brass having various grain sizes are given in Table III.

Specimens with either type of notch (see Figs. 1(*b*) and (*c*)) were loaded as rotating cantilever beams. The data obtained are plotted in the typical *S*-log *N* diagrams shown in Figs. 3 and 4.

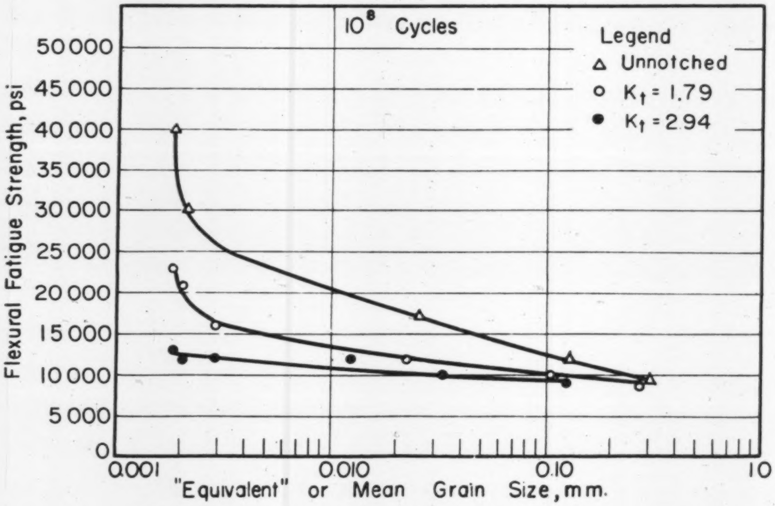


FIG. 6.—Influence of "Equivalent" or Mean Grain Size on the Fatigue Strength at 10^8 Cycles

The fatigue strengths at 100 million cycles for specimens with each notch and for the various grain sizes investigated are listed in Table IV.

For purposes of quantitative comparison, the fatigue strength for any given number of stress cycles can be obtained from the curves of Figs. 3 and 4 for each grain size investigated. These values have been replotted as a function of material grain size in Figs. 5 and 6 for various numbers of stress cycles. From these curves it can be seen that the fatigue strength of the material at each selected life increased as the grain diameter was reduced.

By scaling data from the smooth curves of Figs. 5 and 6, the fatigue strength reduction factors, K_f, were computed for each grain size at each selected life, and the values listed in Table V. From a comparison of these data, it can be seen that for a given life, K_f increased as the grain size decreased. It is also important to note that for any one grain size, K_f in general was *slightly larger* when high loads were employed to produce failure at lower numbers of cycles. This is contrary to the popular belief that higher average stresses cause a localized redistribution of stress that may lower the strength reduction factor.

The fatigue strength reduction factors, K_f (as computed from the curves shown in Fig. 6) and the fatigue notch-sensitivities, q, are plotted *versus* the grain size of the brass in Figs. 7 and 8. It can be seen from these illustrations that K_f and q are not unique material constants but are dependent upon both the grain size of the metal and the notch root radius. In addition, it can be observed that as the grain diameter is reduced, K_f and

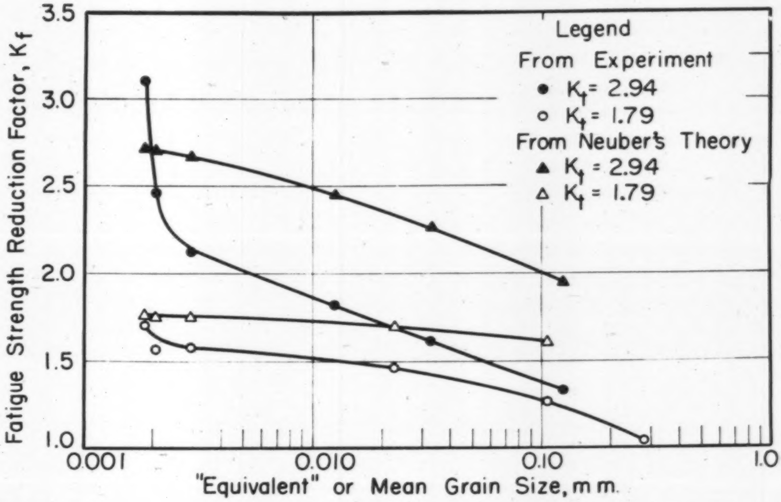


FIG. 7.—Fatigue Strength Reduction Factors at 10^8 Cycles Compared with Neuber's Theory of Notched Material Behavior.

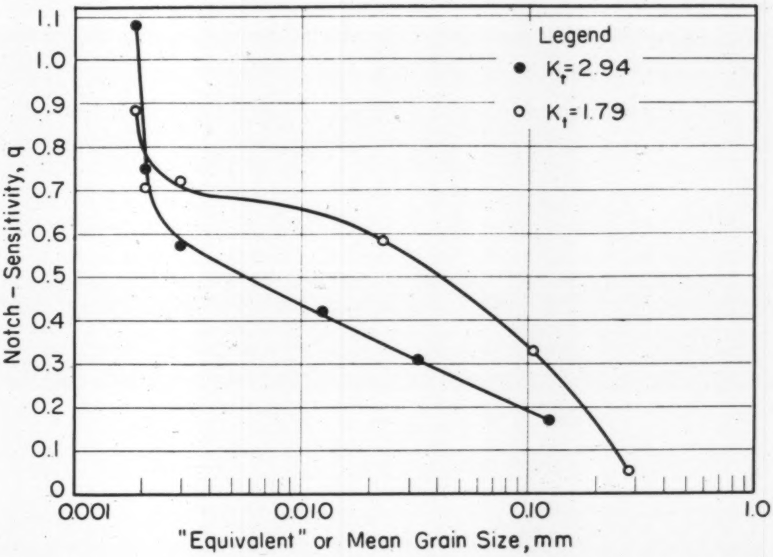


FIG. 8.—Influence of "Equivalent" or Mean Grain Size on Notch Sensitivity at 10^8 Cycles.

q for both notches more nearly approach the values predicted by the theoretical stress concentration factors, K_t. This behavior is in agreement with that observed by Peterson (1, 2). The stress gradient set up in the specimen by the notch, results in an appreciable stress drop across any grain located at the root of the notch. There would be a greater drop in stress across a large grain (and hence the average stress would be smaller) than for a small grain size. For the same maximum (or peak) stress, a fine grain located in the critical zone is therefore subjected to a more severe condition of stressing.

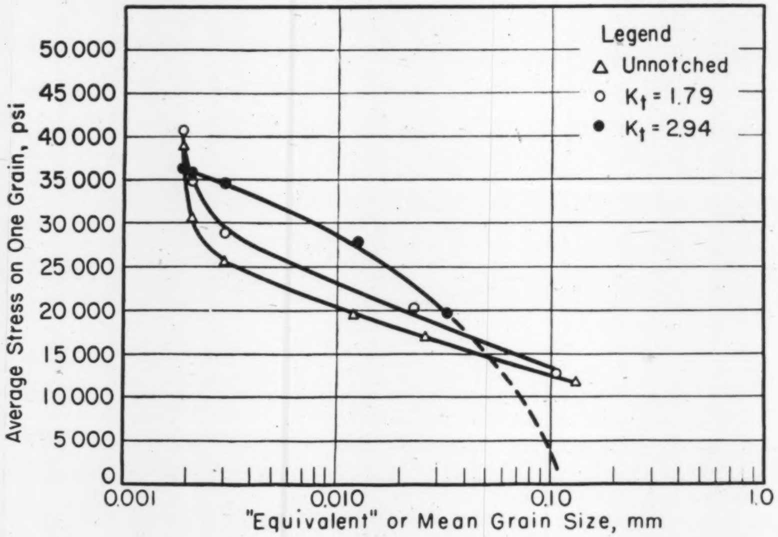


FIG. 9.—Average Stress on One Grain Located in the Critically Stressed Surface of a Notched or Unnotched Specimen (Based on 10^8 Cycles of Stress).

It would be of considerable aid in formulating a design procedure if a rational basis for predicting K_f and q was available. The results of Neuber's (3) analysis of the effect of an elementary particle on notched material behavior, for the condition of static loading, were investigated for their applicability to this problem. When the surface curvature of a member is relatively sharp, the effective stress concentration factor, according to Neuber, is given by the following expression:

$$K_f = 1 + \frac{K_t - 1}{1 + \sqrt{\frac{\rho'}{r}}} \qquad (1)$$

where:

K_f and K_t = previously defined,
r = notch radius, and
ρ' = half the width of the elementary particle.

Neuber also found that for relatively sharper notches, the notch flank angle, ω, had to be considered. The effect of the notch flank angle was found to be satisfied by the following expression:

$$K_f = 1 + \frac{K_t - 1}{1 + \frac{\pi}{\pi - \omega}\sqrt{\frac{\rho'}{r}}} \qquad (2)$$

The liberty of using fatigue symbols in Neuber's expressions has been taken since his static theory was applied to the case of repeated loading.

If the grain diameter of the brass is assumed to be the same as Neuber's concept of an elementary particle, Eqs 1 and 2 yield the curves shown in Fig. 7 for 100 million cycles of stress. The values of K_f for both notches as calculated from Fig. 6 have also been included for comparison. One-half the grain diameter was assumed to be equal to ρ', which is half the width of Neuber's elementary particle. Equation 2 was used for the notch having a root radius of 0.005 in. and a flank angle of 60 deg, whereas Eq 1 was applied for the notch having a root radius of 0.020 in. From the curves in Fig. 7 it can be seen that Neuber's theory predicts fatigue strength reduction factors which are considerably higher than the experimental values for all but the smallest grain sizes considered. Further, it can be seen that there is a greater difference between the theoretical and experimental values for the notch having a root radius of 0.005 in. than the results for the notch with a root radius of 0.020 in. It must be remembered, however, that Neuber considered the material to be subjected to static loads.

AVERAGE STRESS ON CRITICALLY STRESSED GRAIN

It was thought that some correlation might exist between the average stress on a grain located in the surface of an unnotched specimen and that acting on a grain located at the root of the notch of a notched specimen. The stress gradient in the unnotched specimen was determined from the elementary flexure formula. The stress gradients set up by the two notches were calculated from an expression approximated by Seely and Smith (**14**) from Neuber's work. This expression is as follows:

$$\frac{S}{K_t S_n} = \frac{2.5}{\rho} + \frac{1}{c} \quad \ldots\ldots\ldots\ldots (3)$$

where:

S = maximum stress gradient, psi per in.,

$K_t S_n$ = theoretical maximum stress, where S_n is the nominal stress as determined by elementary flexure formula, psi,

ρ = notch root radius, in., and

c = radius of section at root of notch, in.

This expression yields the maximum stress gradient at the root of the notch of a shallow-grooved bar under bending. The criterion for a groove being shallow is that the depth of the groove be less than one-half the radius of the net section at the notch.

The average stress on a grain located at the root of either notch was computed in the following manner. The maximum stress on the grain was assumed to be equal to the product of the nominal fatigue strength (for that particular grain size) and the theoretical stress concentration factor, K_t. The maximum stress gradient in the specimen was computed for the given notch (by Eq 3) and the decrease in stress across the grain was approximated by multiplying the grain diameter by the stress gradient. The minimum stress on the grain could thus be approximated by taking the difference between the maximum stress and this decrease in stress; the approximate average stress was then computed by taking one-half the sum of the maximum and minimum stresses on the grain. In each case the fatigue strength for a particular grain size was obtained from the smooth curves of Fig. 6.

The average stress on a grain located at the root of the notch is compared in Fig. 9 with the average stress on a grain in the surface of an unnotched specimen for each grain size investigated. It can be seen in Fig. 9 that the average stresses on grains in the unnotched specimens and in the specimens having a theoretical concentration factor K_t = 1.79 are in rather close agreement for all grain sizes considered. However, in the case of the notch having a root radius of 0.005 in. (K_t = 2.94), considerable deviation is observed for grain sizes greater than about 0.03 mm. This small root radius sets up an extremely steep stress gradient over a very shallow depth below the root of the notch, and the slope to the stress distribution curve changes markedly across the specimen. Thus for the larger grain sizes, the arbitrary assumption of a constant stress gradient across one grain is greatly in error. For example, with the largest grain diameter, 0.125 mm, the decrease in stress across the grain (as predicted by assuming a constant stress gradient) is so large that a large compressive stress would be necessary at the bottom of the grain. Hence, for this sharp notch, the stress gradient as calculated from Eq 3, in the manner described above, introduces considerable error in calculating the values for the average stresses on the larger grain sizes.

CONCLUSIONS

By means of flexural fatigue tests the notch sensitivity of a typical single-phase metal was investigated for a wide range of grain sizes and for specimens with two different stress raisers. As a result of this study, the following conclusions seem justified:

1. A *decrease* in the grain size (or of "equivalent grain diameter" of cold-rolled brass) resulted in the following observed change in properties:

   (*a*) The fatigue strength of both notched and unnotched specimens was increased.

   (*b*) For notched specimens the fatigue strength reduction factor. K_f, and the notch-sensitivity, q, were both increased.

   (*c*) For the finest grain sizes studied the values of K_f and q tended to approach the values predicted by the theoretical stress concentration factors, K_t.

2. In general, for any given grain size, the fatigue strength reduction factor and the notch-sensitivity were slightly *larger* for the higher stress levels that produced failure at low values of fatigue life.

3. The fatigue strength reduction factor and notch sensitivity are *not* unique material constants; their values depend upon both the notch root radius and the grain size of the metal.

4. A fair correlation was shown to exist between the calculated *average* stress on the most highly stressed grain in a notched specimen with that calculated for an unnotched specimen of the same grain size.

Acknowledgment:

This investigation was conducted in the research laboratories of the Department of Theoretical and Applied Mechanics as part of the work of the Experiment Station, University of Illinois, in cooperation with the Office of Naval Research, United States Navy, under Contract N6ori-071(04). Acknowledgment is due to G. M. Sinclair and W. J. Craig for making available the results of a portion of their investigations and for their many helpful suggestions during the course of the work.

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DISCUSSION

Mr. A. B. Wilder[1] *(presented in written form)*.—A number of years ago H. F. Moore and A. B. Wilder directed experimental studies at the University of Illinois on the influence of grain size on the mechanical properties of several steels. The work was conducted by Messrs. Roy Lorentz, Jr. and Harlan Oehler and was never published. In reviewing the paper by Messrs. R. W. Karry and T. J. Dolan, the thought occurred that a summary of some of the results may be of interest in comparing 70-30 brass with steel.

In the studies conducted by Messrs. Lorentz and Oehler, the variations in measured grain size were obtained by heat treatment followed by slow cooling in a furnace. The fatigue specimens were 0.250 in. in diameter and the notched specimens had a 0.02-in. radius at the root of the notch. The tension impact specimens were 0.200 in. in diameter and the notched specimens had a 0.02-in. radius at the root of the notch. A standard Charpy V-notch specimen was used. The results are summarized in the accompanying Table VI. It will be observed that the endurance limit of unnotched specimens and the ratio of unnotched to notched endurance limit increased with decreasing grain size. The endurance limit of the notched specimens was not appreciably changed with different grain sizes. Other properties shown in Table VI are of general interest.

Preliminary results indicated that lower carbon steels behave in a manner similar to AISI 1045 and 1050 steels; however, results obtained with quenched-and-tempered AISI 2345 steel indicated that quenching and tempering treatments resulting in different grain sizes may influence the endurance limit of notched specimens.

TABLE VI.—MECHANICAL PROPERTIES.

ASTM Grain Size	Endurance Limit			Tension Impact			Charpy Impact, ft-lb	Tensile Strength, psi
	Unnotched, psi	Notched, psi	Ratio, Unnotched to Notched	Unnotched, ft-lb	Notched, ft-lb	Ratio, Unnotched to Notched		
				AISI 1045				
0	35 000	19 000	1.8	59.0	42.0	1.4	10.0	85 700
4	35 000	19 000	1.8	66.0	49.0	1.3	9.0	84 500
7	38 000	19 000	2.0	66.0	49.0	1.4	17.0	85 900
				AISI 1050				
0	30 000	22 000	1.4	86.0	34.0	2.6	4.0	94 300
2	32 000	20 000	1.6	84.0	35.0	2.3	5.0	92 500
7	36 000	21 000	1.7	73.0	41.0	1.7	14.0	92 300
				AISI 2345				
−1	47 500	21 500	2.2	90.0	45.0	2.0	15.0	114 400
2	52 000	22 000	2.4	101.0	52.0	1.9	23.0	113 800
7	54 500	20 000	2.7	112.0	54.0	2.1	25.0	110 000

Mr. Horace Grover[2].—Studies of the influence of grain size on fatigue notch-sensitivity of metals and alloys are much to be desired. The authors are to be commended in carrying out work along this important line.

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[1] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

[2] Division Chief, Battelle Memorial Inst., Columbus, Ohio.

Although it is pointed out in the paper that grain sizes were not actually measured in the cold-rolled brass, the meaning of the "equivalent" grain sizes used in the analysis is not discussed in great detail. Discussion of the physical meaning of this equivalent grain size in the reference paper by Sinclair and Craig[3] is also limited. It would seem that extrapolation of hardness *versus* mean grain-diameter curves from the region of annealed materials into the region of cold-worked materials warrants very careful consideration of the possible differences in the nature of grains developed in these different ways. Such differences may be very important in regard to fatigue notch-sensitivity. More extensive discussion of this point by the authors would be very helpful.

Mr. R. E. Peterson[4] (*by letter*).—This paper represents an important contribution in the field of notch effect in fatigue; the choice of a simple single-phase metal in which the grain size could be controlled is the key idea on which the contribution is based.

In general one would expect $q = 1$ to be approached asymptotically as the grain size is decreased. The sharp upturn (Fig. 7), with q going above 1.0, seems unusual and I would like to ask if the authors have an explanation of this.

Since the notch involves biaxial stress, it may be of interest to consider what direction the results take if the Mises criterion is assumed. It turns out that for the Mises criterion the q curves of Fig. 7 would be raised and, except for the above-mentioned "up-turn," would approach closer to 1.0. With our present state of knowledge, it is not certain what procedure is best so that the foregoing observation should be regarded as speculative.

The direction taken by the investigation reported in this paper is an interesting one and it is hoped that this work will be continued.

Mr. T. J. Dolan (*author's closure*).—The authors wish to thank Mr. Wilder for his contribution of additional data on the effect of grain size in steels. In preparing the paper, we had no intention of inferring that this analysis and experimental data would apply to complex metals such as steels and their alloys in which the hardening constituents are widely dispersed. The strengths of these complex metals are more dependent upon the shape and distribution of the various hardening constituents dispersed within the grains. The purpose in selecting alpha brass in this case was to use a simple, single-phase metal in which the strength of the grains was dependent primarily on their size (in an inverse manner such that the smaller the grain, the stronger its resistance to plastic deformation).

We are rather strongly of the opinion that for steel or similar alloys, perhaps it is not grain size that is important but some mean free path for slip in the grain, as determined by the spacing of carbides and other hardening constituents. The approach used was considered of a fundamental type, to see whether for the simple, single-phase metal there was some relationship between fatigue strength and grain size.

Mr. Grover suggests further interpretation or discussion of the meaning of the "equivalent" grain size employed for the cold-worked brass. As he points out, there may be questions raised about the validity of the extrapolation in relating grain size to hardness measurements alone. However in spite of the existence of many arbitrary methods of measuring various types of grain size (and correspondingly many different opinions as to which grain size is most important) there is no standardized procedure available for determining a "grain size" for cold-worked metal. Therefore, we adopted the procedure used by Sinclair and Craig[3] who made rather extensive studies of the same heat of alpha brass used by the authors. Sinclair and Craig found a close correlation between the "equivalent" grain size as determined by hardness measurements and the standard mechanical properties of tensile strength, fatigue strength, strain hardening exponent, etc.

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[3] G. M. Sinclair and W. J. Craig, "Influence of Grain Size on Work Hardening and Fatigue Characteristics of Alpha Brass," *Transactions*, Am. Soc. Metals, Vol. 44, p. 929 (1952).

[4] Manager, Mechanics Department, Westinghouse Research Laboratories, East Pittsburgh, Pa.

In general, it was felt that if grain size is a controlling factor in affecting strength properties, the converse might also be true; that is, that an approximate measure of effective grain size could be obtained from a test such as the hardness test which measures resistance to deformation of the metal. In the absence of an accepted standard for grain size of cold-worked metal, this method seemed to offer a rational and useful measure that could be used for the brass as a criterion for practical engineering purposes.

With regard to the symbol ρ' in Neuber's equation, we did not employ the value Neuber had suggested for certain metals but examined his equation by assuming that perhaps the grain itself was the elementary structural unit. Thus by using Neuber's equations with ρ' equal to half the grain size the computed technical strength reduction factors were plotted in Fig. 8 where they were compared with the experimental strength reduction factors. They seem to follow the same general trends, but the computed Neuber values were somewhat higher in strength reduction factor than the experimental values. Perhaps this would indicate that the structural unit of importance is not the grain but something corresponding to a part of a grain, or group of grains. However Neuber's analysis is based upon a theoretical approach for an elastic condition, and here it is applied to a fatigue condition for which the assumptions might not be strictly applicable.

The authors have no complete explanation of the sharp upturn in values of notch sensitivity index q noted by Mr. Peterson in Fig. 7 of the paper. The values of q are quite sensitive to small experimental errors since they involve a ratio of two numbers each of which may vary appreciably with small changes in experimental data. It seems possible that the fatigue strength for unnotched specimens at the smallest grain size may have been slightly higher than would normally be experienced from the trend of the other data. This would partially account for the sharp increase in values of q for the smallest grain size employed. However since the notch sensitivity index is affected by several geometrical factors and is not an inherent property of the metal, the authors did not wish to attach much importance to the data in Fig. 7. The fact that the q for the smallest grain size exceeded a value of 1.0 by a small amount in one case is not significant and may be within the limits of experimental error.

STATIC AND FATIGUE PROPERTIES OF CARBON, SILICON, AND HIGH-STRENGTH LOW-ALLOY STEEL PLATES HAVING A HOLE*

By Frank Baron[1] and Edward W. Larson, Jr.[1]

Synopsis

The purposes of this investigation were to determine the fatigue strengths of various high-strength low-alloy steels, the relationship, if any, between the fatigue strengths and the stress-strain characteristics of the steels, and the relative advantages of the various steels for resisting fatigue loadings. Fifteen different plate steels were considered, including 7 low-alloy steels, 4 silicon steels, 3 ordinary carbon steels, and 1 rimmed steel. The test specimens were long and narrow and had a $1\frac{1}{16}$-in. hole at the center. Several ranges of zero to tension cycles of loading were considered for each kind of steel. For many of the fatigue specimens, measurements were made of the strains at the edges of the holes. Strain concentration factors were determined for the various ranges of loading.

The number of cycles to failure for the various kinds of steels were related to the average stresses and to the maximum longitudinal tensile strains at the net-sections of the specimens. For the type of specimen considered, the fatigue strengths of the various steels increased with the tensile strengths and with the yield strengths. The fatigue lives of the various steels were more closely related to the maximum strains than to the average stresses or to the maximum stresses at the net-sections of the specimens.

Differences in view exist in the technical literature concerning the relative advantages which high-strength structural steels may have over carbon structural steels for resisting fatigue loads. The differences in view are a consequence of the results reported for small-scale notched and unnotched specimens as compared to the results reported for unpolished plates with a hole or for riveted joints. Several investigators (**1, 2, 3**)[2] have reported that the fatigue strengths and endurance limits of small, polished specimens are directly related to the tensile strengths of the materials. In 1935, Kommerell (**4**) reported that the ratio of the fatigue strength to the tensile strength of a high-strength low-alloy steel can be decreased by the introduction of a stress-raiser such as a hole. Graf (**5**) reported the same conclusion with particular reference to the results of fatigue tests of riveted joints. However, in 1938, Wilson (**6**) and Thomas reported that the decrease in the above ratio was so great for unpolished plates with a hole and for riveted joints that the fatigue strengths of a silicon and of a nickel steel were essentially the same as those of a carbon steel. Consequently, many engineers concluded that the advantages which high-strength structural steels might have over carbon steels for resisting static loads could not be realized for fatigue loads. Other engineers believed that the data reported by Wilson and Thomas were insufficient to warrant such a conclusion.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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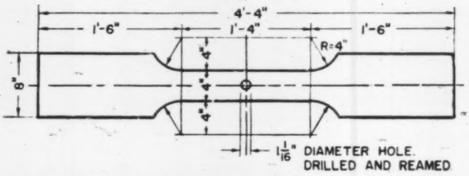
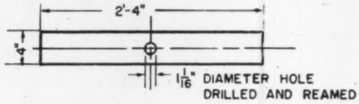
[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 823.

The above differences in view led to an investigation to determine whether the fatigue characteristics of different steels were related to the static strengths of the steels when tested in the form of a plate with a hole. A study was also made to determine the relationship between the fatigue lives and the maximum longitudinal tensile strains for the different steels. The study was based on the concept advanced by the metallurgist or research worker that fatigue cracks develop as a consequence of the minute deformations that occur within the crystalline structure of a material. The various attempts to relate fatigue lives with stresses have deemphasized the important rôle which deformations play in determining the fatigue life of a member. For this reason the authors believed that deformations or strains should provide a better basis than stresses for evaluating the effects of different variables on the fatigue lives of members. This belief was strengthened when it was realized that, for the various ranges of fatigue loading considered, the strains in the vicinity of the discontinuity exceeded the elastic limits of the steels.



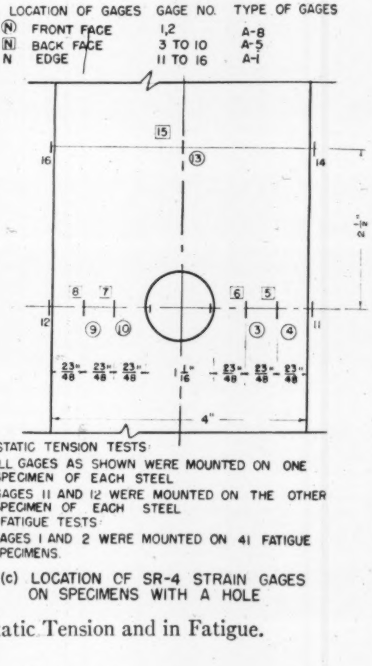
NOMINAL THICKNESS OF PLATE = 5/8"
NOMINAL AREA OF NET-SECTION = 1.84 SQ. IN.
NOMINAL AREA OF GROSS-SECTION = 2.50 SQ. IN.

(a) DIMENSIONS OF FATIGUE SPECIMENS



NOMINAL THICKNESS OF PLATE = 5/8"
NOMINAL AREA OF NET-SECTION = 1.84 SQ. IN.
NOMINAL AREA OF GROSS SECTION = 2.50 SQ. IN.

(b) DIMENSIONS OF STATIC TENSION SPECIMENS



NOTE: FOR STATIC TENSION TESTS:
ALL GAGES AS SHOWN WERE MOUNTED ON ONE SPECIMEN OF EACH STEEL
GAGES 11 AND 12 WERE MOUNTED ON THE OTHER SPECIMEN OF EACH STEEL
FOR FATIGUE TESTS:
GAGES 1 AND 2 WERE MOUNTED ON 41 FATIGUE SPECIMENS.

(c) LOCATION OF SR-4 STRAIN GAGES ON SPECIMENS WITH A HOLE

FIG. 1.—Dimensions of Specimens Tested in Static Tension and in Fatigue.

DESCRIPTION OF TEST SPECIMENS

Fifteen steels, ranging in tensile strengths and having the mill-scale left intact, were selected for the program reported herein. Fatigue tests and static tension tests were made of long narrow plates having a hole in the center. Several ranges of zero to tension cycles of loading were considered for each kind of steel. The dimensions and details of the specimens tested in static tension and in fatigue are shown in Fig. 1. The hole in the center of each specimen had a final diameter of $1\frac{1}{16}$ in. and was drilled and reamed in the laboratory. The selected ratio of width of specimen to diameter of hole was 3.76. For this ratio, a stress-concentration factor of 2.32 is obtained by the theory of elasticity. In this report, stress-concentration factor is defined as the ratio of the maximum stress at the edge of the hole to the average stress on the net-section of the specimen. For each kind of steel, all fatigue specimens, static tension specimens, and coupon specimens were obtained from a single plate. Since only one sample plate was obtained for each kind of steel, the results do not necessarily represent the maximum, the minimum, or the average result which should be expected for each grade of steel. In order to insure that unwarranted comparisons are not made on the basis of trade designations, the various steels are designated by letters A through O.

In general, the fatigue and static tension specimens were machine flame-cut $\frac{1}{2}$ in. oversize and were then machined to the final dimensions. However, two of the four groups of ASTM Specification A 94[3] silicon steel specimens were prepared by machine flame-cutting to size and then post-heating the edges. These groups of specimens were designated by N and O. Each group of post-heated specimens was prepared by the manufacturer of the steel. All other fatigue and static tension specimens were machined by a single large fabricator.

TABLE I.—MECHANICAL PROPERTIES OF THE VARIOUS STEELS.[a]

Kind of Steel	Material	Yield Strength for an Offset of 0.2 per cent, psi	Tensile Strength, psi	Elongation, per cent		Reduction in Area, per cent	Roughnesses of Plate Surfaces[b]	Average Hardness of Material[c]
				8-in. Gage Length	2-in. Gage Length			
High-strength low-alloy (ASTM A 242)	A	55 000	79 500	27.2	50.0	59.3	very rough	84
	B	44 400	65 200	30.2	55.0	61.2	very smooth	73
	C	47 800	82 600	25.8	47.8	60.5	rough	85
	D	57 000	74 400	26.4	50.0	60.1	very rough and pitted	84
	E	48 200	77 900	27.9	50.2	61.5	smooth	81
	F	49 500	78 800	27.8	50.0	60.8	rough	81
	G	51 200	73 600	29.6	51.0	60.6	rough	80
Rimmed	H	31 100	52 700	33.4	57.5	63.8	smooth	52
Carbon (ASTM A 7)	I	36 600	70 000	28.8	50.5	53.3	very rough	72
	J	34 400	62 100	31.0	53.0	57.2	rough	66
	K	35 400	60 000	30.3	54.2	60.5	very rough	65
Silicon (ASTM A 94)	L	51 800	90 000	23.4	40.5	44.2	rough	83
	M	48 400	88 800	23.4	43.5	50.4	very rough	83
	N	51 500	90 800	22.9	40.5	46.9	fairly smooth	85
	O	46 500	83 600	25.7	49.2	58.6	very rough	84

[a] Average properties for two coupon specimens per steel. The average modulus of elasticity for the various steels ranged between 29,400,000 and 31,500,000 psi.

[b] Fatigue specimens.

[c] Average of six readings per steel. Rockwell B Scale, 100 kg load, $\frac{1}{16}$ in. steel ball.

STATIC AND RELATED TESTS OF THE VARIOUS STEELS

Various types of tests were made of each material. These included static tests of coupon specimens, hardness surveys, and micrographic studies. The tests were needed for interpreting the results of the fatigue tests and of the static tension tests of the specimens with a hole.

For each material, two coupon specimens were tested in static tension to determine the mechanical properties and the stress-strain characteristics. The average properties are given in Table I and a typical stress-strain curve for each material is given in Fig. 2. The coupon specimens had machined edges, standard rectangular cross-sections, and 8-in. gage lengths. The tests were made in a Baldwin-Southwark machine having a capacity of 120,000 lb. The strains were measured with a Riehle dial extensometer having an 8-in. gage length. Two sequences of loading were considered. One sequence consisted of increasing the load in successive increments; however, at certain intervals the load was decreased to zero to obtain the permanent set for each interval of loading. For each case of unloading, the ratio of the decrease in stress to the decrease in strain was a constant and was equal to the initial modulus of elasticity. The other sequence of loading consisted of increasing the load in successive increments until failure occurred. For each sequence of loading, the increment of load was 2000 lb before excessive yielding of the specimen occurred and 5000 lb thereafter. The mechanical properties of each steel were essentially the same for both sequences of loading.

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[3] Tentative Specifications for Structural Silicon Steel (A 94 - 50 T), 1952 Book of ASTM Standards, Part 1, p. 376.

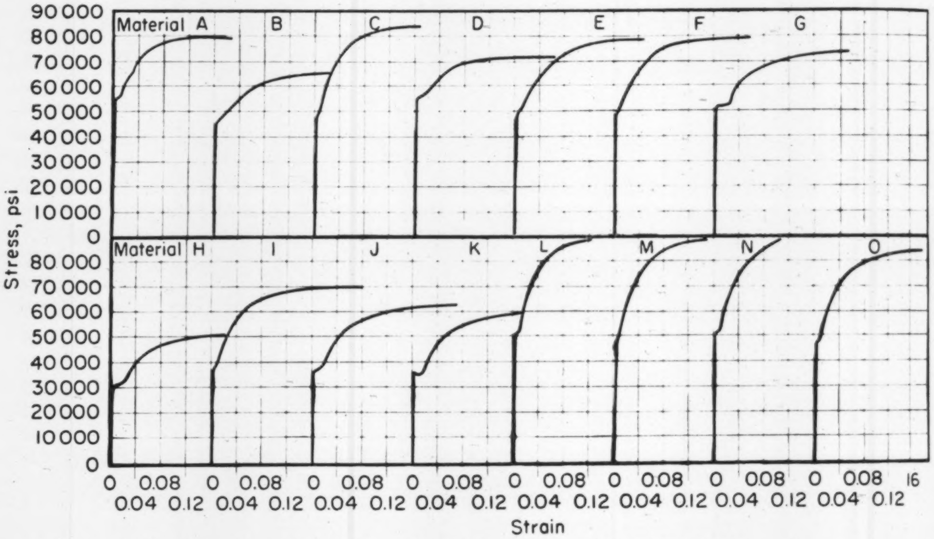

FIG. 2.—Typical Stress-Strain Curves for the Various Steels.

Inspection of Table I shows that the three carbon steels, I, J, and K, met the tension requirements of the ASTM Specification A 7[4] and the four silicon steels, L to O, met the tension requirements of ASTM Specification A94. ASTM Specification A 242[5] was applicable for the high-strength low-alloy steels, except for material B which had an average tensile strength of 65,200 psi. The yield strengths for the low-alloy steels were greater than for the carbon steels and were about the same as for the silicon steels. For the low-alloy steels, the yield strengths were between 58 and 77 per cent of the tensile strengths. For the silicon and the carbon steels, the yield strengths were between 52 per cent and 59 per cent of the tensile strengths. The ductilities of the low-alloy steels were about the same as for the carbon steels and were appreciably greater than those for the silicon steels.

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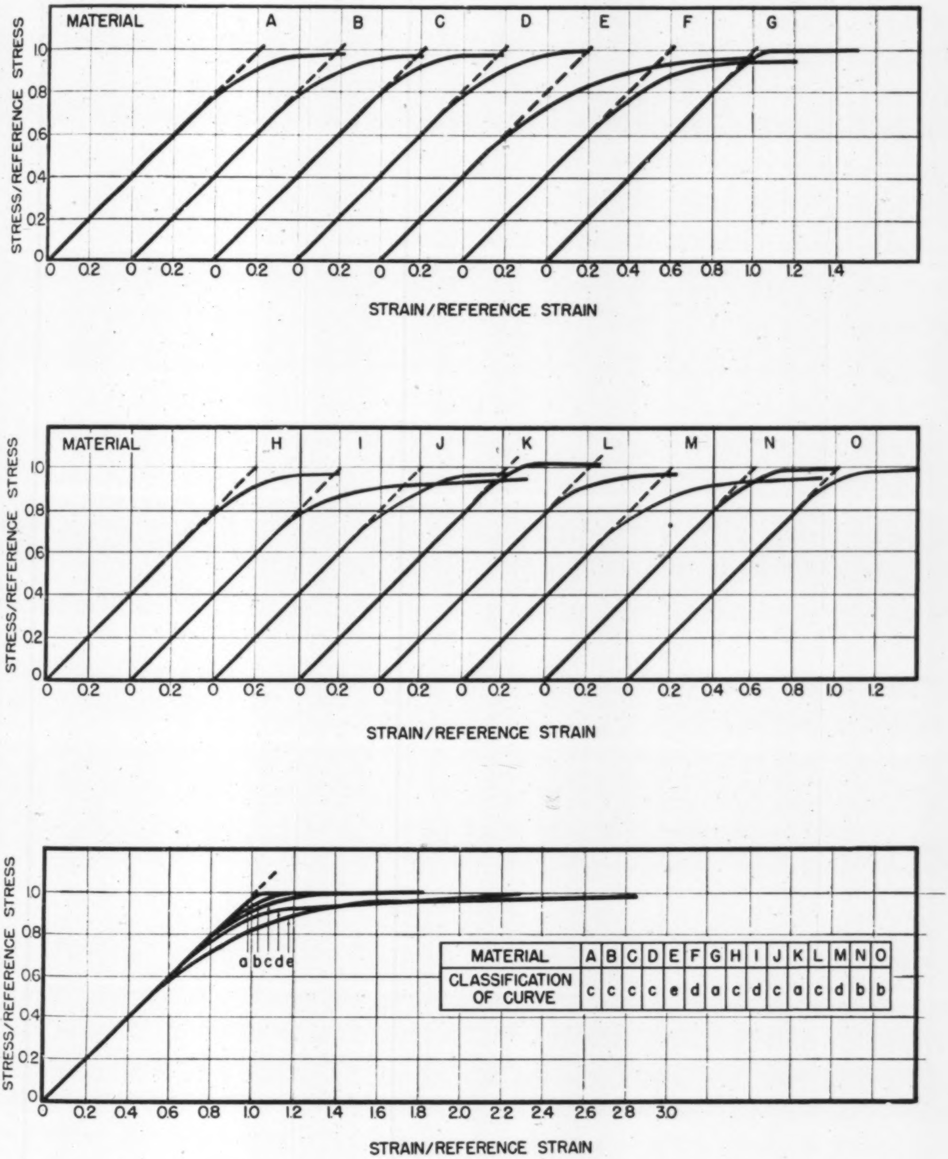
[4] Tentative Specifications for Steel for Bridges and Buildings (A 7 – 52 T), 1952 Book of ASTM Standards, Part 1, p. 356.

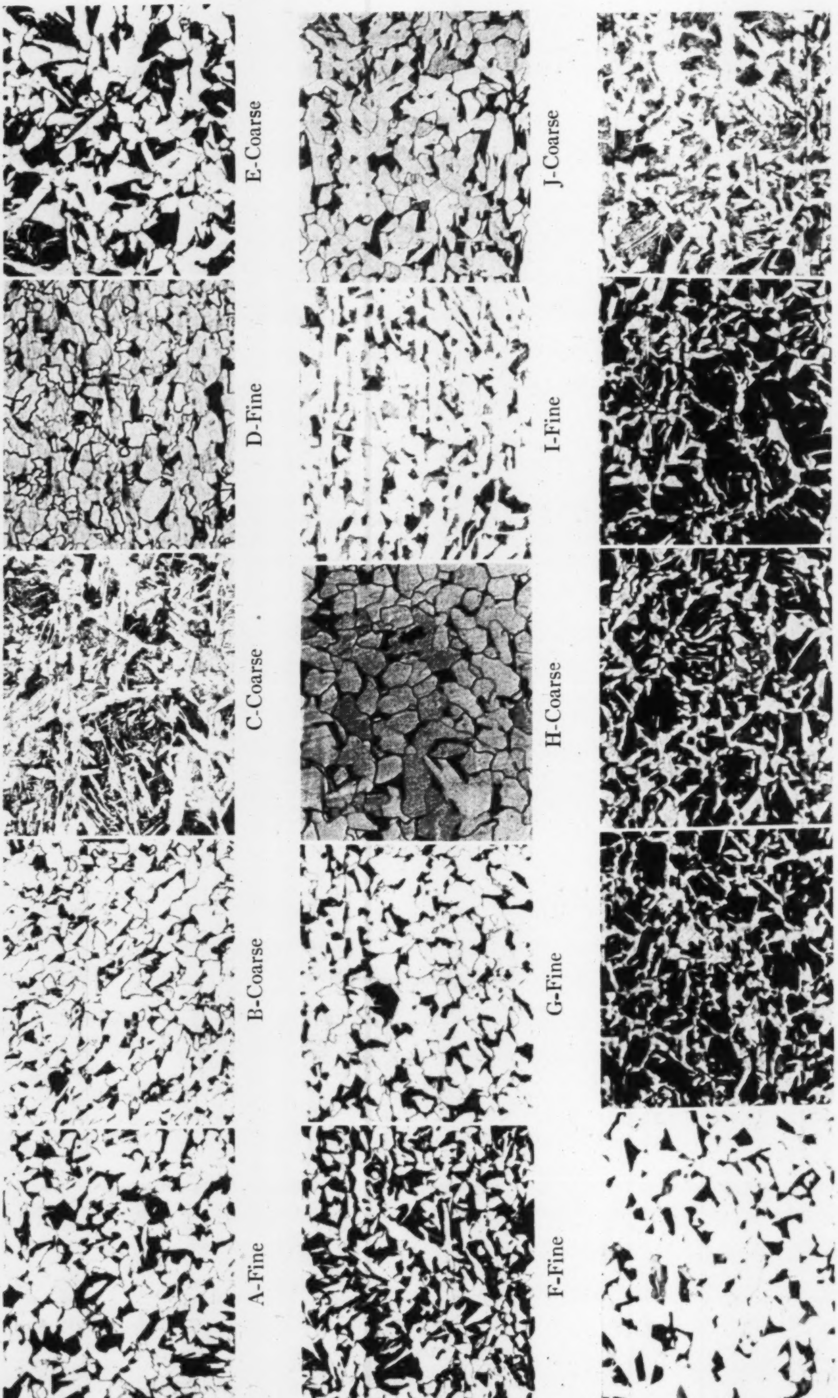
[5] Tentative Specifications for Low-Alloy Structural Steel (A 242 – 52 T), 1952 Book of ASTM Standards, Part 1, p. 437.

The introduction of a new steel for structural purposes immediately raises questions concerning the mechanical properties and the stress-strain characteristics of that steel. Such questions arise as to what is the shape of the stress-strain curve, how is yield point or an equivalent term to be defined, and how is the yield point to be determined in acceptance testing. Manufacturers of steel frequently use a testing machine which produces an autographic stress-strain



MATERIAL	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
CLASSIFICATION OF CURVE	c	c	c	c	e	d	a	c	d	c	a	c	d	b	b

FIG. 3.—A Comparison of the Shapes of the Various Stress-Strain Curves.



A-Fine B-Coarse C-Coarse D-Fine E-Coarse

F-Fine G-Fine H-Coarse I-Fine J-Coarse

K-Coarse L-Coarse M-Fine N-Fine O-Coarse

FIG. 4.—Microstructures of the Various Steels.

McQuaid-Ehn grain size, fine or coarse.

record but will not indicate the existence of a yield point. The contract requirements for reporting a yield point cannot be fulfilled because a "drop of beam" or "halt in gage" cannot be observed by such a procedure of testing. For this reason, it has been suggested that the unit stress corresponding to a unit strain of 0.005 be accepted and reported as an equivalent yield point.

For the above reasons, dimensionless diagrams were drawn for comparing the shapes of the various stress-strain curves. These diagrams are shown in Fig. 3. For each steel, the ratio of stress to a reference stress was plotted *versus* the ratio of strain to a reference strain. The reference stress was different for each steel and was selected to be the stress corresponding to a strain of 0.005. The reference strain was also different for each steel. In selecting each reference strain, the initial tangent to the stress-strain curve was extended until it intersected the value of the reference stress. The strain which corresponded to this point of intersection was taken as the reference strain. The deviations of each stress-strain curve from the initial tangent were summarized and each stress-strain curve was classified in one of five groups: *a* to *e*. All five shapes of stress-strain curves were obtained for the low-alloy steels. Shapes *a*, *c*, and *d* were obtained for the three carbon steels and shapes *b*, *c*, and *d* for the silicon steels. The classification is given in Fig. 3.

For each steel, a comparison was made of the yield strengths determined in different ways. Yield strength was defined as the stress obtained by the 0.2 per cent offset method and the stresses corresponding to strains of 0.002 and 0.005. For several steels, the yield points were indicated by the drop of the beam. The yield strengths obtained by the 0.2 per cent offset method were almost the same as those obtained for a strain of 0.005. As a result of the different shapes that were obtained for the stress-strain curves, the yield strengths obtained for a strain of 0.002 were at times appreciably less than those obtained for a strain of 0.005. For this reason, the authors believe that if yield strength is to be reported as the unit stress corresponding to a selected value of unit strain, the selected unit strain should be about 0.0025 for the types of steels considered herein.

Rockwell hardness tests were made of each steel and the results are given in Table I. These tests were made of samples cut from the original plates. For each material, hardness surveys were also made to determine whether the flame-cutting operations had influenced the properties of the finished specimens. These surveys were made across the widths of several specimens which had been tested in fatigue. Thirteen readings were taken across the width of each specimen and the averages of these readings were about the same as those given in Table I. No appreciable variation in hardness existed across the width of each specimen. This indicated that the flame-cutting operations had little, if any, influence on the properties of the specimens with machined edges.

Micrographs showing the internal structures of the various steels were made to determine whether the differences in the fatigue strengths of the various steels could be related to the McQuaid-Ehn grain sizes and the actual microstructures of the steels. The micrographs and the McQuaid-Ehn grain sizes are given in Fig. 4. An examination was also made of the fatigue specimens to determine the surface roughness of each steel. An indication of the relative roughnesses of the various steels is given in Table I. Six degrees of roughness are indicated and ranged from a very smooth surface with mill-scale intact to a very rough surface with little mill-scale but with the parent steel pitted and rusted.

STATIC TENSION TESTS OF SPECIMENS WITH A HOLE

For each material, two static tension tests were made of the specimens with a hole. One specimen of each material was subjected to a continuously increasing load until failure occurred. This specimen had a $\frac{1}{4}$-in. square grid scribed on a surface and a strain gage located on each edge to indicate when yielding occurred across the entire net-section. The other specimen of each material had 16 SR-4 strain gages attached to it and was subjected to a different sequence of loading. This sequence consisted of an increase in load until a load of 50,900 lb was reached. The load was then decreased to a value of 10,000 lb, and then increased until failure occurred. Strain gage readings were taken at 2000-lb increments or decrements of load until yielding occurred across the entire net-section. The location of each strain gage is shown in Fig. 1. It should be observed that only longitudinal tensile strains were measured. The effects of the curvature of the hole and of the transverse strains on the SR-4 strain gage readings were neglected.

TABLE II.—TENSION TESTS OF THE SPECIMENS HAVING A $1\frac{1}{16}$-IN. HOLE.[a]

Kind of Steel	Material	Approximate Yield Load, lb	Approximate Yield Stress, psi[b]	Ultimate Load, lb	Ultimate Stress, psi[b]
High-strength low-alloy (ASTM A 242)	A	102 500	55 800	148 800	80 900
	B	85 000	46 300	126 600	68 900
	C	95 000	51 700	157 200	85 600
	D	105 000	57 200	138 500	75 400
	E	87 500	47 600	149 400	81 400
	F	90 000	49 000	145 100	79 000
	G	90 000	49 000	138 900	75 500
Rimmed	H	57 500	31 300	99 100	54 000
Carbon (ASTM A 7)	I	70 000	38 100	133 800	72 800
	J	65 000	35 400	116 700	63 600
	K	65 000	35 400	113 500	62 000
Silicon (ASTM A 94)	L	95 000	51 700	169 900	92 700
	M[c]	90 000	49 000	166 000	90 400
	N[d, e]	105 000	57 200	167 000	91 200
	O[e]	95 000	51 700	159 700	87 100

[a] Average of two specimens per steel.

[b] Based on a nominal area of 1.836 sq in. The actual areas did not differ by more than 1 per cent from the nominal area.

[c] One specimen had a 100 per cent brittle fracture.

[d] One specimen had a 50 per cent brittle fracture.

[e] Specimens with post-heated edges.

Table II gives for each steel the average yield load and the average ultimate load. The yield load for a material in the form of a plate with a hole was about the same irrespective of the procedure of loading and was considered to be that load at which yielding had progressed across the entire net-section. The ultimate load for a material was also about the same irrespective of the procedure of loading.

The distributions of longitudinal strains across the net-section and a gross-section were obtained for one specimen of each material. Selected examples of these distributions are given in Fig. 5. The examples were selected to illustrate the influence of the magnitude of the yield point and the shape of the stress-strain curve on the distributions of the strains. For a given value of load, the strains were largest at the edges of the holes. These strains were about the same for a load of 15,000 lb irrespective of the kind of steel. For such values of load, each material behaved elastically and the strains were proportional to load. For strains in the elastic range, the theoretical stress concentration factor was 2.32 as compared to an experimental value of about 2.44. The load for which the maximum strain was equal to the yield strain was different for each material. For high values of load, the maximum strains increased at a greater rate than the increase in load and were related to the stress-strain characteristics of the materials. The maximum strains for loads of about 45,000 lb and over were largest for the steels with the low values of yield point. These strains were greater

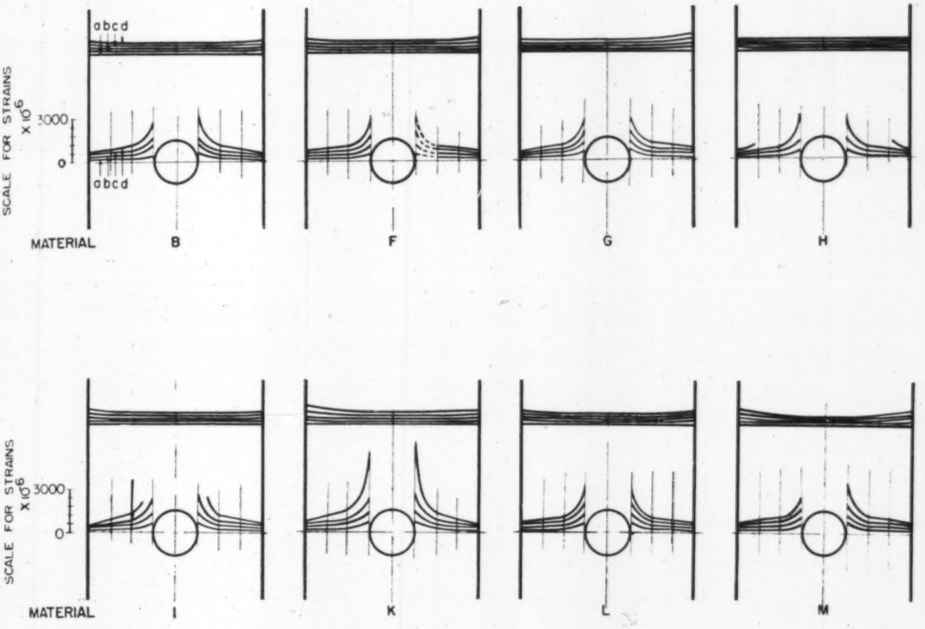


FIG. 5.—Examples of Distribution of Longitudinal Strains for Static Tension Specimens.

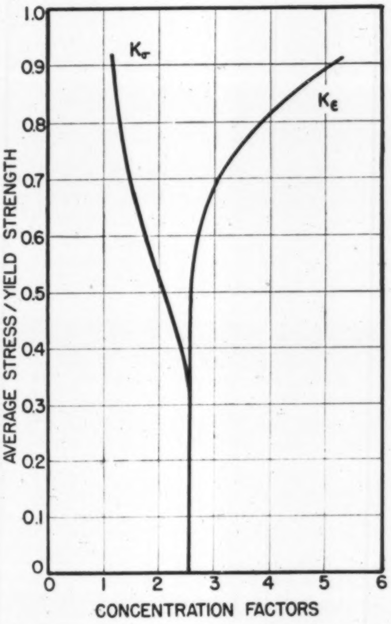


FIG. 6.—Stress and Strain Concentration Factors for Material K.

for the carbon steels than for the low-alloy and the silicon steels.

Stress-concentration factors, K_σ for members loaded in different ways and having different types of discontinuities have been reported upon by various investigators. These factors were determined analytically (**7**) as well as photoelastically (**8**). Strain concentration factors for strains in the inelastic range have been studied less extensively and have just recently been reported upon in the technical literature (**9, 10, 11, 12**). For the program reported herein, strain concentration factors, K_ϵ, were determined for various values of average stress on the net-sections of the specimens. The strain concentration factor was defined as the ratio of the maximum strain at the edge of the hole to the strain of the coupon specimen for the same average stress on the net-section of each specimen. Curves were drawn relating the strain concentration factors with average stresses on the net-sections and with ratios of average stress to yield strength (0.2 per cent offset). A typical example of the latter curves is shown in Fig. 6. The corresponding relationship for stress concentration factors is given in the same figure. It was observed that when the maximum strain exceeded the proportional limit of a material the strain concentration factors increased with an increase in load, whereas the stress concentration factors decreased. For a load of 30,000 lb or less, the strain concentration factors were a constant for each material and were approximately the same for each kind of steel. The strain concentration factors began to increase at a different value of load for each material. The curves relating the strain-concentration factors with the ratios of average stress to yield strength had approximately the same shape for each kind of steel except for material A and for the silicon steels with post-heated edges. The curves for the former group of steels indicated that in the inelastic range the strains at the edge of the hole were principally influenced by the magnitude of the yield point for each steel. The curves for the silicon steels with the post-heated edges indicated that the residual compressive strains reduced the tensile strains caused by tension loads. The curve for material A indicated that the strain at the edge of the hole exceeded the yield strain at a lower value of load than was to be expected. This may have been caused by other factors than those which have been considered.

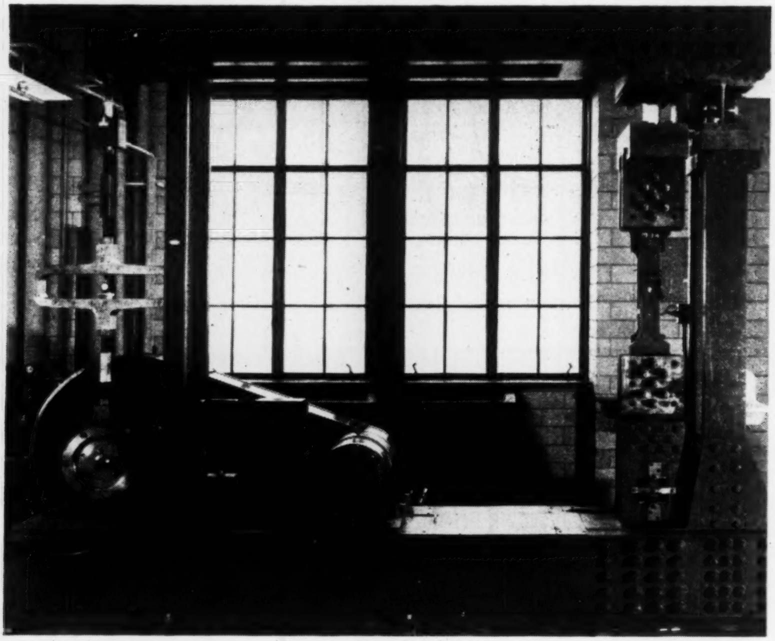


FIG. 7.—80,000-lb Capacity Fatigue Machine.

FATIGUE TESTS OF SPECIMENS WITH A HOLE

All fatigue tests were conducted in the 80,000-lb capacity fatigue machine shown in Fig. 7. The frequency of load-

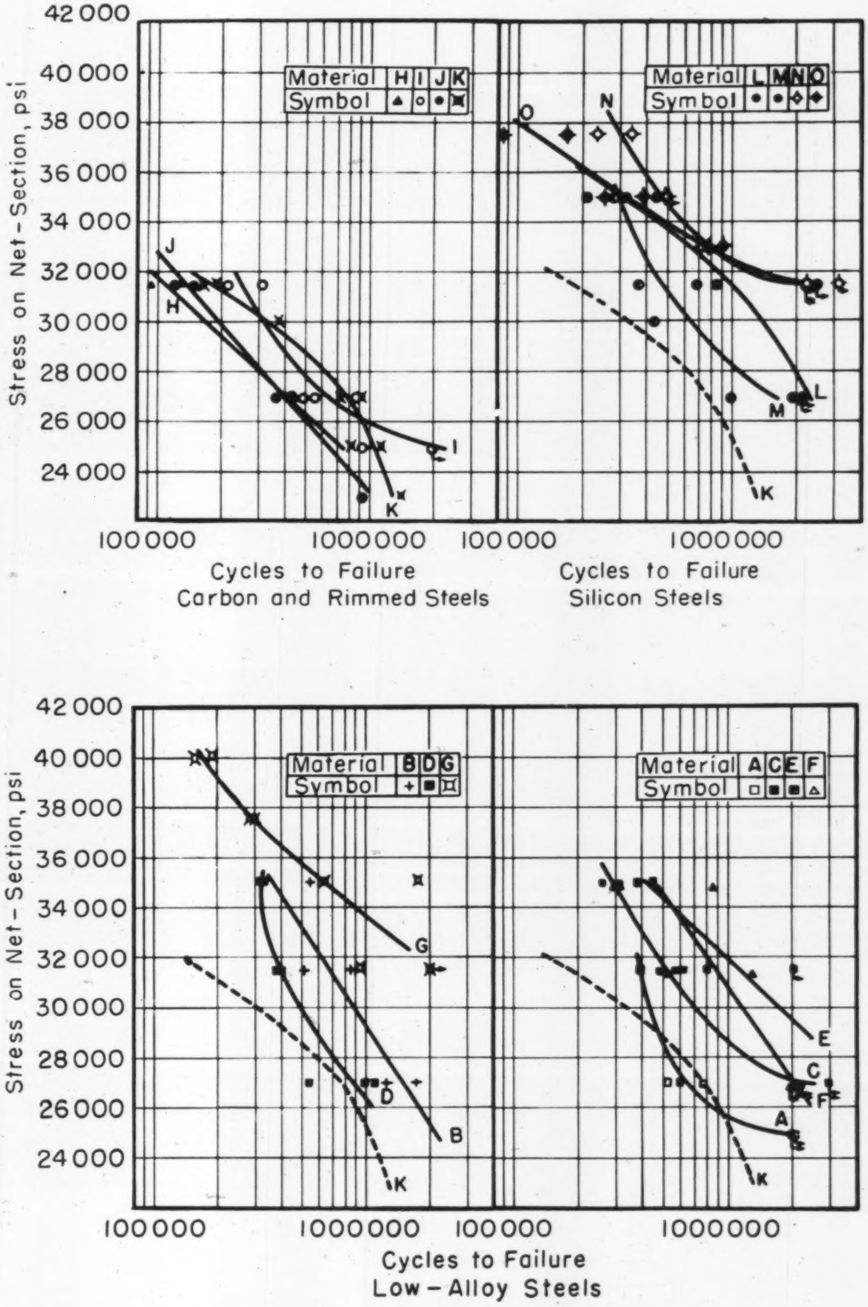


FIG. 8.—Average Stress *versus* Number of Cycles to Failure.

ing was approximately 200 cycles per min. The load on a specimen was indicated by a dynamometer which had been calibrated for static loads. The dynamometer was checked for the normal frequency of the machine. During each fatigue test, the load on the specimen was checked at least every 12 hr. A limit switch was provided to shut off the machine when a specimen failed.

A total of 109 specimens were tested in fatigue. In general, at least 7 specimens of each material were tested in zero to tension cycles of loading. Three or more ranges of loading were considered for each steel, except for the rimmed steel. Duplicate specimens were usually tested at each range of loading. When the results of duplicate specimens differed appreciably, a third specimen was tested

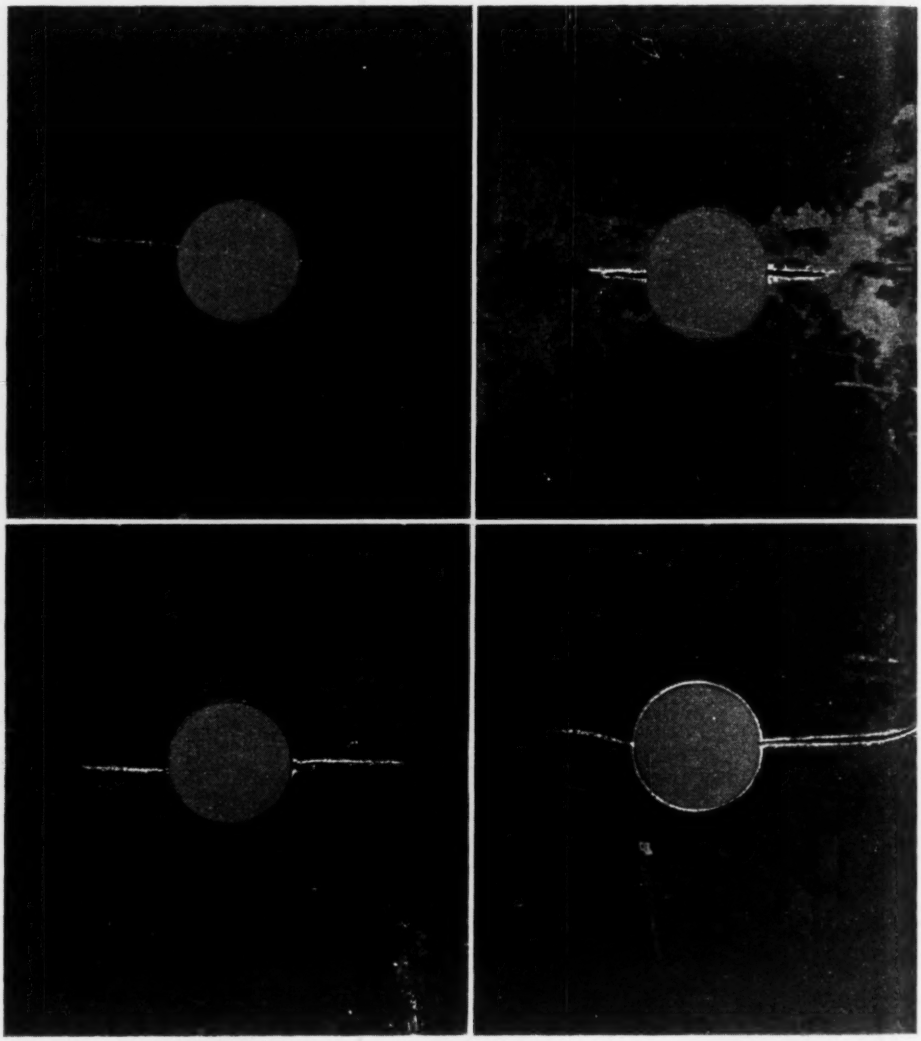


FIG. 9.—Typical Fractures of Fatigue Specimens.

at the same range of loading. Each steel was tested at a 0 to +31,500 psi range of stress. The other ranges of stress differed for each material and depended on the results obtained for the preceding ranges of loading. For 12 of the 15 steels, a 0 to +27,000 psi range of stress was also considered. In general, all specimens were

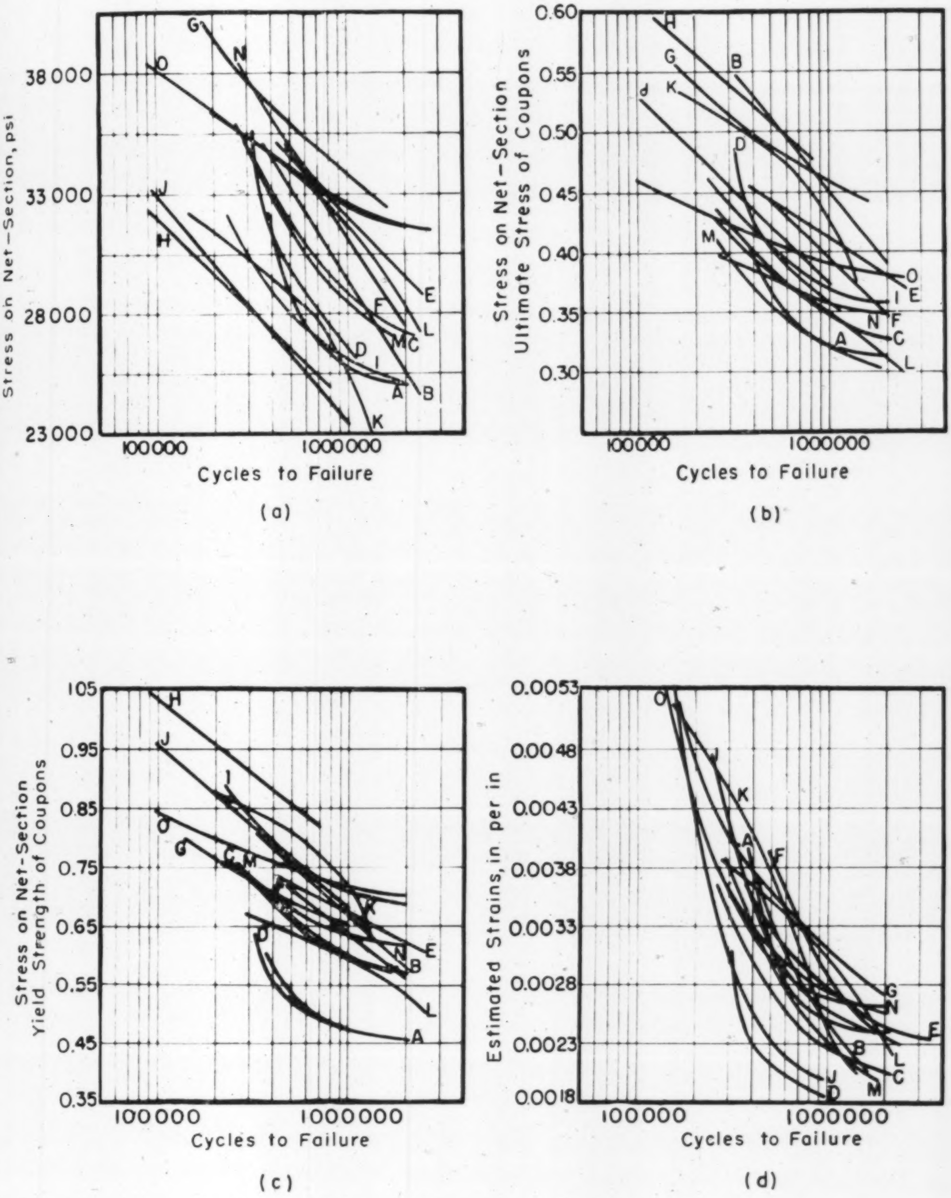


FIG. 10.—Summary of Curves Relating Average Fatigue Lives with Stresses, Ratios of Stresses and Estimated Strains.

subjected to a continuous cycle of loading except for the very short intervals of time when the load was being checked.

The results of the fatigue tests are given in Fig. 8. For each test, the average stress on the net-section was plotted *versus* the number of cycles to failure. For each material, an estimate was made of a single curve considered to be the best fit for the data obtained. In general each curve was drawn through the computed average of the fatigue lives obtained at each value of average stress. The curves of Fig. 8 are grouped in accordance with the kinds of steel and the tensile strengths. Two groups are given for the low-alloy steels. A tensile strength of 75,000 psi was selected for distinguishing between the latter groups of curves.

No difficulties were encountered in defining failure of the various specimens except for some of the post-heated specimens. For the latter specimens, N and O, the cracks slowly progressed across the widths of the specimens indicating that residual compressive strains existed in the central portion of these specimens due to the post-heating operations. Additional evidence that such strains existed was obtained from the measurements of strains taken during the static tension tests and the fatigue tests. In the vicinity of a hole, the residual compressive strains reduced the tensile strains caused by tension loads. Typical fatigue cracks for the different types of steels are shown in Fig. 9.

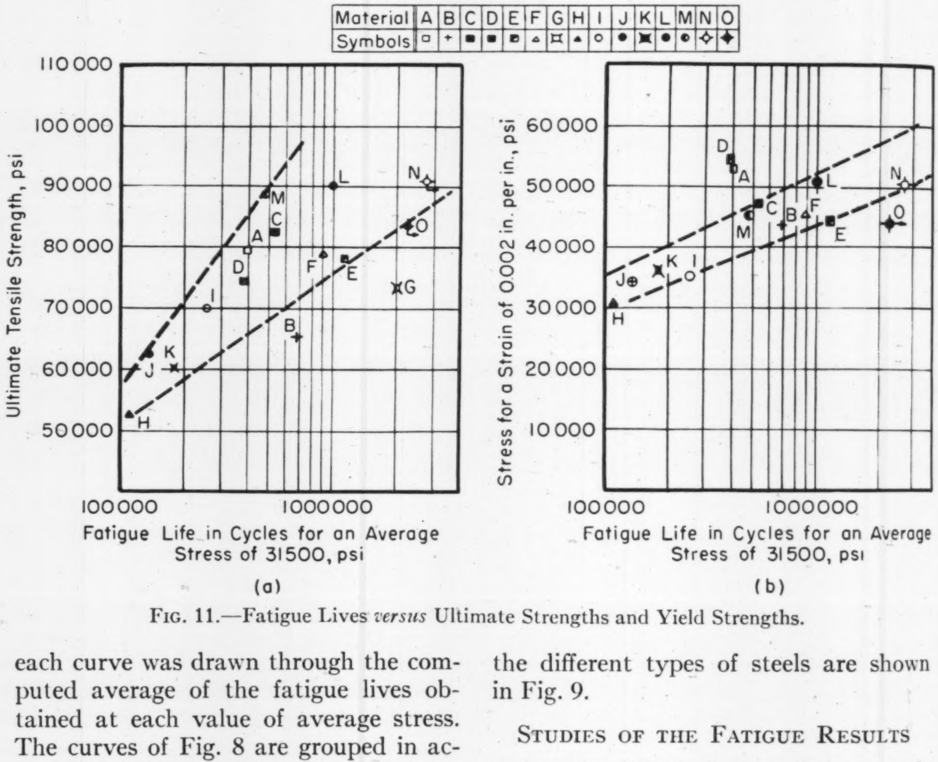


FIG. 11.—Fatigue Lives *versus* Ultimate Strengths and Yield Strengths.

STUDIES OF THE FATIGUE RESULTS

A study of the fatigue results was made to determine the relative advantages of the various steels for resisting fatigue loadings and to determine whether the fatigue strengths were related to the mechanical properties or the stress-strain characteristics of the steels. A study was also made to determine whether the fatigue lives of the various steels were related to the maximum strains at the edges of the holes. The results of these studies are summarized in Figs. 10, 11, and 12.

Figure 10(*a*) shows that the fatigue lives for a given stress on the net-section differed for the various steels. The differences in the fatigue lives of the various steels were most marked at the lower ranges of loading. The average fatigue life for a given stress on the net-section was less for each of the carbon steels than for any of the other steels except A and D. It is believed that the fatigue strengths of A and D were influenced by the surface roughnesses of these steels. The surfaces were very rough as compared to those of the other steels. The average fatigue lives for a given stress on the net-section were greater for the silicon steels with post-heated edges than for the silicon steels with machined edges. Material G apparently had a marked advantage over the other steels in resisting fatigue loadings.

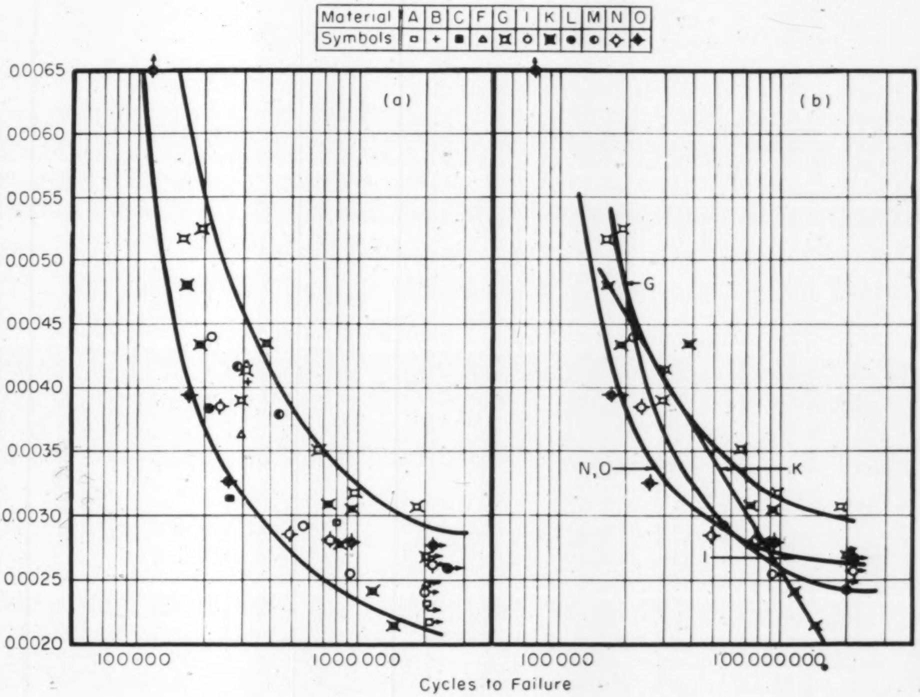


FIG. 12.—Maximum Strains *versus* Cycles to Failure.

Figure 11(*a*) shows the influence of the ultimate strengths on the fatigue lives of the various steels. For each steel, the ultimate tensile strength is plotted *versus* the average fatigue life for a stress of 31,500 psi. The plotted values were obtained from the results of the coupon tests and from the curves drawn in Fig. 10(*a*). A trend was observed in the fatigue characteristics of the various steels. The trend was for the steels with the higher ultimate strengths to have higher fatigue strengths.

Ratios of fatigue strengths to static strengths are plotted in Fig. 10(*b*) for the various fatigue lives of the 15 steels. For failure at 300,000 cycles of loading, the ratios for the various steels ranged between 0.39 and 0.56. The ratios ranged between 0.32 and 0.47 for failure at 1,000,000 cycles of loading.

The relationships between the yield strengths and the fatigue characteristics of the 15 steels are shown in Fig. 11(*b*). For each steel, the yield strength is plotted *versus* the average fatigue life for a stress of 31,500 psi on the net-section. The plotted values were obtained from the stress-strain diagrams and from the *S-N* diagrams. As for the diagram relating fatigue strengths with ultimate strengths, Fig. 11(*b*) shows a trend in the fatigue characteristics of the various steels. The trend was for the steels with the higher yield strengths to have higher fatigue strengths. The plotted values for the various steels, except A and D, were within the band indicated in the figure. On the basis of this figure, it can be stated that the fatigue strengths of the various steels were as closely related to the yield strengths as to the ultimate strengths. Relationships between the fatigue lives and the corresponding ratios of the average fatigue strengths to the yield strengths are given in Fig. 10(*c*). For failure at 300,000 cycles of loading, these ratios ranged between 0.66 and 0.91. The ratios for failure at 1,000,000 cycles of loading ranged between 0.47 and 0.77.

For each specimen tested in fatigue, an estimate was made of the maximum strain at the edge of the hole and was plotted *versus* the fatigue life of the specimen. The maximum strains for the fatigue specimens were assumed to be the same as those obtained for the duplicate specimens tested in static tension. Figure 10(*d*) shows the average relationships which were obtained between the estimated strains and the fatigue lives of the various steels. For the ranges of fatigue loadings considered, the estimated strains exceeded the respective yield strains of the various materials. It is observed that a reasonable estimate of strains could also have been obtained by means of the stress-strain curves of the materials and by means of the strain concentration curve given in Fig. 6. The relationships given in Fig. 10(*d*) indicated that perhaps the fatigue lives of the various specimens were principally related to the maximum strains.

For 41 fatigue specimens, the strains at the edges of the hole were measured by means of SR-4 strain gages. Two gages of the A-8 type were mounted on the inside face of a hole and were located as shown in Fig. 1. For a given range of loading, the strains were measured during the first application of load, the release of this load, and for several succeeding cycles of fatigue loading. For each of these specimens, the maximum strain occurring during the first cycle of loading was plotted *versus* the cycles to failure. In each case, the initial crack occurred on the same side of the hole where the maximum strain was measured. An envelope of the test results is drawn in Fig. 12. Sufficient readings had not been taken to permit a curve to be drawn for each material. However, sufficient readings had been taken to permit curves to be drawn for materials G, I, K, N, and O. These curves are given in Fig. 12 and relate the maximum strains to the corresponding lives of the steels.

A comparison of the strain-*N* curves given in Fig. 12 with the corresponding stress-*N* curves given in Fig. 10(*a*) shows that the fatigue lives of the various specimens are more closely related to the maximum tensile strains on the net-sections than to the average stresses or the maximum stresses on the net-sections of the specimens. This may be seen by comparing the magnitudes of the deviation of the test data from the curves of averages for each material. A smaller degree of deviation or scatter is obtained when the fatigue lives are plotted *versus* maximum strains rather than *versus* stresses. A study of the curves for materials G, I, K, N, and O shows that the stress-N curves differ considerably from each other whereas the strain-N curves do not differ appreciably for the various materials. In Fig. 12(a), the fatigue lives of all specimens for which strains were measured are plotted *versus* the maximum strains. An envelope of the data is drawn in the same figure. The results of the various specimens represented in Fig. 12(a) are plotted in Fig. 13 on the basis of average stresses rather than on the basis of maximum strains. It is observed that when all of the data irrespective of the kind of steel are plotted on the basis of average stresses, a random distribution of data having no definite trend is obtained. However, when the data are plotted as in Fig. 12(a) on the basis of maximum strains, a relatively narrow band of values having a definite trend is obtained.

For the ranges of fatigue loading considered, the measured strains were not in the elastic range; they were in the plastic range and were directly related to the stress-strain characteristics of the materials. Therefore, the fatigue characteristics of these steels were intimately related to the stress-strain characteristics of the steels in static tension. A comparison of the diagrams based on measured strains to those based on estimated strains shows that the envelopes are practically the same and that the individual curves are almost the same. This would indicate that perhaps a reasonable estimate can be made of the average relationship between the fatigue lives and maximum strains of another type of specimen provided the stress-strain characteristics of the steel and the strain-concentration factors for the given type of specimen are known. An inspection of the envelope given in Fig. 12(a) shows that for the higher values of strain, the width of the envelope is narrow and the slope is steep. It is observed that for values of strain in this steep region, the fatigue lives for the different steels are essentially the same even though the strains are appreciably different.

Other factors than those dealt with in the preceding discussion had an influence on the fatigue characteristics of the different kinds of steels. For example, the surface roughnesses of materials A and D undoubtedly decreased the fatigue strengths of these steels. The residual compressive strains which existed in the post-heated specimens increased the fatigue strengths of the silicon steels. The internal structures of the steels had little influence on the fatigue strengths. The specimens of the steels having the same ultimate strengths but lower fatigue resistances had a fine McQuaid-Ehn

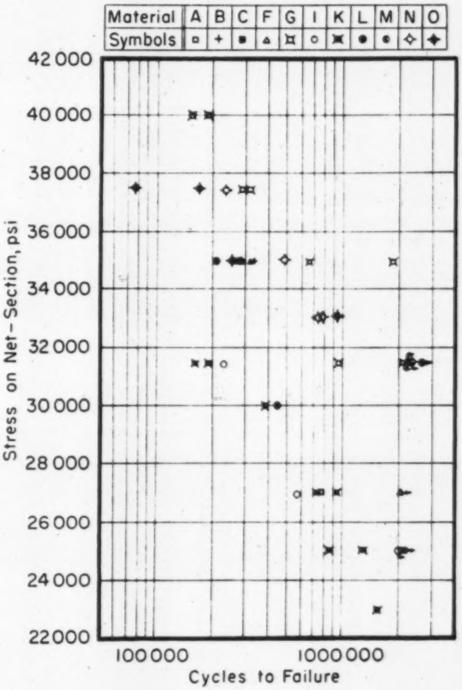


FIG. 13.—Average Stresses *versus* Cycles to Failure.

grain size. The metallographic examination gave no other indication as to why the steels having the same ultimate strengths had different fatigue strengths. No relationship was obtained between the chemical compositions and the fatigue strengths of the different steels.

CONCLUSIONS

On the basis of these tests, the following conclusions can be drawn concerning the fatigue properties of the various steels and the relationships of these properties to certain variables.

1. For the ranges of loading considered, the initial strains at the edges of the hole were in the inelastic range and were dependent on the magnitudes of the yield strengths and on the shapes of the stress-strain curves.

2. The fatigue lives for a given stress on the net-section differed for the various steels. These differences were most marked at the lower ranges of loading. The average fatigue lives for a given stress on the net-section were greater for all of the low-alloy steels, except A and D, than for the carbon steels. The average fatigue lives were also greater for the silicon steels than for the carbon steels. Material G had a marked advantage over the other steels in resisting fatigue loadings.

3. The fatigue lives of the various steels were more closely related to the maximum longitudinal tensile strains at the edges of the holes than to the average stresses or to the maximum stresses on the net-sections of the specimens.

4. Other factors such as the degree of surface roughness and residual strains had a noticeable effect on the fatigue strengths of several steels. The internal structures of the various steels had little effect.

Acknowledgments:

The investigation was conducted in the Department of Civil Engineering at Northwestern University in cooperation with the Research Council on Riveted and Bolted Structural Joints of the Engineering Foundation, Inc. J. L. Beckel, Samuel Epstein, Jonathan Jones, T. H. Lovell, C. Neufeld, N. M. Newmark, E. J. Ruble, W. M. Wilson, B. L. Wood, and the authors served as members of Project Committee 6 of the Research Council. The committee was given the task of investigating "The Fatigue Strength of High-Strength Steel Riveted Joints." The investigation reported herein represents the first phase of this task.

The support of the sponsors of the Research Council and of the manufacturers who supplied steel for the project is gratefully acknowledged. Particular acknowledgments are given to Samuel Epstein and Carl Haupt for conducting the micrographic studies of the various steels.

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DISCUSSION

MR. W. LEIGHTON COLLINS.[1]—Did the authors observe the location of the fatigue cracks relative to the localized regions of yielding that may have occurred?

MR. FRANK BARON (*author*).—Yes. It is interesting that the fatigue lives of the specimens seemed to be identified on the basis of strains. We had taken measurements of the longitudinal strains on both sides of the holes and observed that the strains on the sides of the holes were not the same for each specimen. However, for each specimen the initial crack developed on the side where the measured strain was the largest. We also observed that the yielding occurred in the immediate vicinity of the cracks and developed in advance of the cracks as the cracks progressed across the width of the specimen. The type of specimen selected was such that not much question existed as to the probable location of the maximum strains. The maximum strains should be expected at the net-sections of the specimens, and, in general, the cracks developed at these net-sections.

MR. COLLINS.—Where yielding could be observed, was the crack adjacent to the yielded portion or in it?

MR. BARON.—I would say that the cracks were in the yielded portions. This was most marked as the yielded portions and the cracks developed across the specimens.

MR. B. J. LAZAN.[2]—Were the authors able to observe the initiation and propagation of the fatigue cracks during the test program? If so, how rapidly did the cracks progress after initiation?

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[1] Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.

MR. BARON.—At the beginning of the test program, we spent considerable time trying to observe how quickly a crack would progress across the width of a specimen. We had microswitches to shut off the fatigue machine as soon as a certain slip or total stretch would occur. Fortunately, the arrangement was sufficiently sensitive to shut off the machine before a crack developed across a specimen. For several specimens we also observed the formation of a crack before the crack had developed sufficiently for the machine to be shut off. In general, after a crack was initiated the crack progressed across the width of a specimen in about 10,000 to 25,000 cycles of loading.

However, difficulties were encountered in defining failure for specimens N and O. These groups of specimens were of silicon steel and had machine-cut and post-heated edges. For these specimens a considerable number of cycles, perhaps 1,000,000, was required for a crack to develop sufficiently far across the width of a specimen in order for us to stop the test. In conclusion, the cracks progressed very quickly after they were initiated, except for specimens N and O.

MR. S. EPSTEIN.[3]—This work has shown very clearly that the higher strength steels gave higher fatigue test results. This was true for the specimens in which the holes were left unfilled. Now let us consider the case where the holes are filled with rivets having high clamping power. The unfilled holes acted as notches, as evidenced by the fact that the cracks in the fatigue specimens all started at the holes. After the holes are filled with rivets of high clamping power, however, the rivet holes would be bridged over by the rivet heads so that the holes would no longer act as notches. In this case then, should we not expect the high-strength low-alloy steels to show an even greater advantage over plain carbon steel than was shown in the present tests with the unfilled rivet holes?

MR. BARON.—This may be hoped for but may not be obtained. We may obtain different results for riveted joints than we had obtained for the specimens with a hole. Further, we may obtain still different results for specimens in the form of bolted joints. We already have tested some materials in the form of bolted joints. The joints were fastened with high-strength bolts torqued to a high tension. The joints did not fail after 3,000,000 cycles of a zero to 40,000 psi range of stress on the net-sections of the joints.

MR. PAUL KUHN.[4]—Do the higher strength steels show any advantage in fatigue if they are compared on the basis of stresses adjusted in proportion to the static strength?

MR. BARON.—Ratios of fatigue strengths to static strengths and of fatigue strengths to yield strengths were plotted for the various fatigue lives of the steels. These plots are given in Figs. 10 (*b*) and (*c*), respectively. Interpretations of advantages or disadvantages based on these plots can be misleading for design purposes. In the design of structures fabricated of the various steels different tension:shear:bearing ratios would probably be selected for the various steels. This could influence the proportioning of members and result in different dead load to live load ratios for the various kinds of steels. Consequently, the relative advantages of the various steels would be different for various structural types and service conditions.

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[3] Research Engineer, Bethlehem Steel Co., Bethlehem, Pa.

[4] Assistant Chief, Structures Research Division, NACA, Langley Field, Va.

ELEVATED TEMPERATURE FATIGUE PROPERTIES OF SAE 4340 STEEL*

By W. J. Trapp[1] and R. T. Schwartz[1]

Synopsis

For steel there is little published information on the effect of mean stress on the axial loading fatigue properties and on the fatigue notch sensitivity and dynamic creep effects at elevated temperatures. These properties of SAE 4340 steel were determined over the temperature range from room temperature to 1000 F.

Material and Procedure

The test material used was rolled $1\frac{1}{8}$-in. diameter bars, all from the same heat of SAE 4340 aircraft quality steel, conforming to Military Specification MIL-S-5000A, with the following analysis:

Carbon, per cent	0.414
Manganese, per cent	0.79
Phosphorus, per cent	0.024
Sulfur, per cent	0.014
Silicon, per cent	0.29
Nickel, per cent	1.76
Chromium, per cent	0.77
Molybdenum, per cent	0.27

Cylindrical unnotched fatigue specimens were rough machined within $\frac{1}{16}$ in. of the finished dimensions and heat treated as follows: 1575 F for $1\frac{1}{2}$ hr, quenched in oil, tempered at 1150 F for $1\frac{1}{2}$ hr and air cooled. It is realized that, even for about the same hardness, treatments for SAE 4340 steel other than quenched and tempered may produce better properties at the higher temperatures, such as about 1000 F and higher. The specimens were given as nearly identical heat treatments as possible. Although there were several batches because of the limited capacity of the furnace, the hardness produced was within two points Rockwell C for all specimens. After heat treatment, the unnotched specimens were turned to 0.400 in. diameter in the gage length of $1\frac{3}{4}$ in. with 1 in.-14 NF-3 threaded gripping ends (Fig. 1(*A*)). The threads were ground to aid in proper alignment of the specimens in the fatigue machine. All tool marks in the gage section of the specimens were removed by hand polishing. A surface finish of about 10 microinches was produced. The notched fatigue specimens were similarly heat treated and machined, except for the gage section, which was turned to 0.450-in. diameter and in which was machined a 60 deg circumferential groove, 0.025 in. deep with a 0.01-in. radius at the bottom (Fig. 1(*B*)). The groove was not polished since microscopic examination showed the surface finish of the groove to be comparable to that of the unnotched specimens. The tension and the creep-rupture unnotched and notched specimens were exactly the same as the fatigue specimens, except for the threaded ends for gripping, which were smaller.

Fatigue tests were conducted in axial loading at room temperature, 600, 800,

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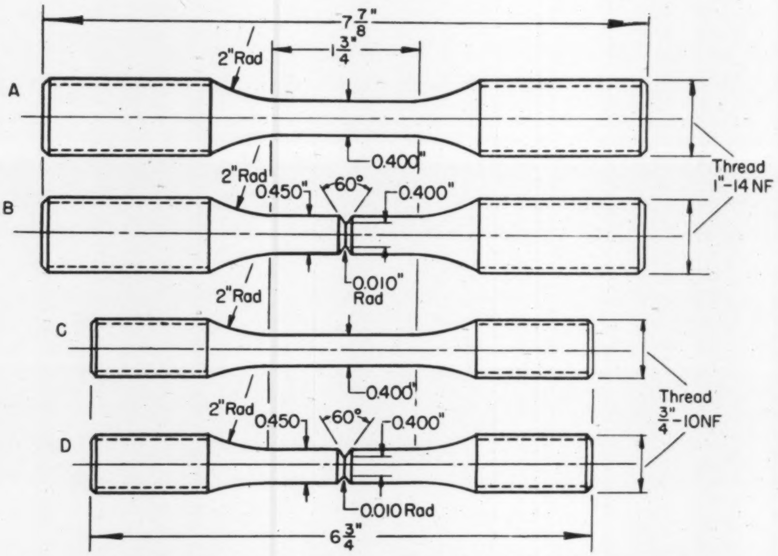
* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Materials Laboratory, Directorate of Research, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.


7 7/8"
2" Rad
1 3/4"
A
0.400"
Thread
1"-14 NF
2" Rad
0.450"
60°
0.400"
B
0.010"
Rad
2" Rad
C
0.400"
Thread
3/4"-10NF
2" Rad
0.450
60°
0.400"
D
0.010 Rad
6 3/4"

A and *B* = Fatigue and stress-rupture. *C* and *D* = Creep rupture.
FIG. 1.—Test Specimens.

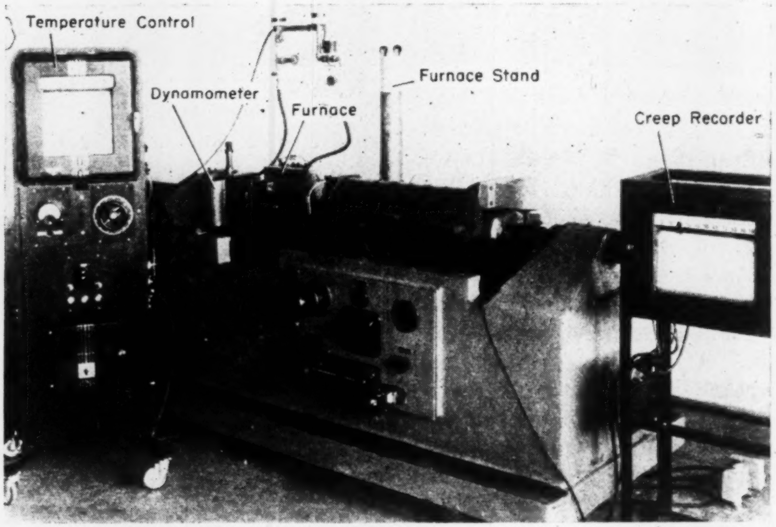

Temperature Control
Dynamometer
Furnace
Furnace Stand
Creep Recorder

FIG. 2.—Equipment for Elevated Temperature Fatigue Testing.

and 1000 F in a 20-ton Schenck fatigue testing machine[2] (Fig. 2). The frequency of the cyclic loading was in a small range of frequency slightly above 2000 cycles per minute. This range of frequency is due to the fact that for this type fatigue machine the amplitude of the cyclic load is controlled by varying over a small range the speed of an eccentric which excites the dynamic loading spring. The specimen grips were made of relatively low heat-conducting material, Inconel X. They were water cooled in order to avoid the influence of temperature on the dynamometer, loading spring system, and the creep measurements. The specimens were fitted with lock nuts, which were processed very carefully in cutting the thread and facing the ends as accurately as possible, so that negligible bending stresses would be applied to the specimen by tightening the nuts.

TABLE I.—TENSILE, CREEP-RUPTURE, AND FATIGUE PROPERTIES OF SAE 4340 STEEL, UNNOTCHED SPECIMENS.

	Tensile Properties					Axial Loading Fatgue Strength at 15×10^6 Cycles				
								Other Stress Ratios		
Temperature, deg Fahr	Tensile Strength, psi	Yield Strength, 0.2 per cent Offset, psi	Elongation in 4 Diam., per cent	Reduction of Area, per cent	Creep-Rupture Strength, at 120 hr, psi	Tension 0 to Max ($A = 1$), psi	Completely Reversed ($A = \infty$), psi	A	Mean Stress, psi	Alternating Stress, psi
Room.........	158 500	146 900	15.0	52.4	...	+115 000	±70 000	0.25	+120 000	±25 000
600..........	152 800	121 400	19.3	59.0	145 000	+96 000	±64 000	0.50	+80 000	±40 000
800..........	125 000	101 500	16.3	67.9	104 000	+82 000	±60 000	0.32	+95 000	±30 000
1000..........	80 700	62 600	20.6	78.6	40 000	+65 000	±39 000	0.50	+40 000	±20 000

TABLE II.—TENSILE, CREEP-RUPTURE, AND FATIGUE PROPERTIES OF SAE 4340 STEEL, NOTCHED SPECIMENS.

	Tensile Properties			Creep-Rupture Strength at 120 hr		Axial Loading Fatigue Strengths at 15×10^6 Cycles						
								Fatigue Strength Reduction Factor, K_f		Other Stress Ratios		
Temperature, deg Fahr	Ultimate Strength, psi	Reduction, of Area, per cent	Static Notch Factor	Creep-Rupture Strength, psi	Creep Notch Factor	Tension 0 to Max ($A = 1$), psi	Completely Reversed ($A = \infty$), psi	Tension 0 to Max ($A = 1$)	Completely Reversed ($A = \infty$)	A	Mean Stress, psi	Alternating, Stress, psi
Room.....	190 000	11.2	0.83	...	...	+57 500	±30 000	2.0	2.3	0.04	+150 000	±6 500
600.....	176 000	17.2	0.87	173 000	0.84	+43 000	±25 000	2.2	2.6	0.10	+120 000	±12 500
800......	154 900	24.3	0.81	120 500	0.87	+49 000	±28 000	1.7	2.1	0.26	+74 000	±19 000
1000......	98 400	33.2	0.82	33 000	1.21	+37 500	±26 000	1.4	1.5	0.50	+28 000	±14 000

The stress distribution in the gage section of each specimen was checked statically with Huggenberger extensometers before starting each test. The maximum deviation from the average strain was found not to be higher than 2 per cent. A more thorough check was conducted with SR-4 strain gages on a specimen picked at random. Three gages were mounted at each end of the gage section on its periphery 120 degrees apart. The maximum deviation in the periphery of one end was found to be 1.4 per cent, in the other end 1.6 per cent. The difference between the average stresses in the peripheries of the two ends was found to be 0.7 per cent. The furnace was a split type provided with three separate heating coils in order to control temperature distribution in the axial direction. The temperature gradient over the gage length of the specimen was maintained within ±5 F at 1000 F; the horizontal arrangement of the furnace in the fatigue machine aids in attaining a uniform temperature. The temperature control thermocouple was fixed to the specimen itself. In this way, the desired temperature was maintained closely, and temperature change in the specimen—for instance, that due to heat generation by damping in the material—was balanced out without overshooting. A Leeds and Northrup Micromax recording controller controlled the testing temperature with an accuracy of ±2 F.

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[2] "Six Ton Schenck Fatigue Testing Machine," Air Force Technical Report No. 5623, August 15, 1947.

TABLE III.—CREEP-RUPTURE DATA.

Unnotched Specimens						Notched Specimens				
Specimen	Temperature, deg Fahr	Rupture Stress, psi	Rupture Time, hr	Elongation in 4 Diam., per cent	Reduction of Area, per cent	Specimen	Temperature, deg Fahr	Rupture Stress, psi	Rupture Time, hr	Reduction of Area, per cent
No. 1	600	144 000	176.5	15.0	53.2	No. 10	600	172 000	200.0	11.7
No. 2	600	146 500	35.7	15.5	57.2	No. 11	600	185 500	0.25	14.0
No. 3	600	151 000	0.25	17.5	61.3	No. 12	600	175 000	39.0	11.9
No. 4	800	101 800	181.8	20.5	64.2	No. 13	800	120 000	100.0	25.7
No. 5	800	109 000	30.5	22.6	71.9	No. 14	800	126 000	42.0	25.5
No. 6	800	118 000	2.3	16.8	71.7	No. 15	800	140 000	2.3	27.0
No. 7	1000	43 000	84.5	32.4	30.2	No. 16	1000	35 000	65.0	6.4
No. 8	1000	46 000	17.8	42.0	55.5	No. 17	1000	50 000	9.3	8.2
No. 9	1000	52 000	9.0	31.5	...	No. 18	1000	60 000	5.5	8.3

The dynamic creep measurement was accomplished by measuring the gripping head travel. The movement of the head was transferred to a leaf spring from which the deflection was picked up by wire gages, amplified, and recorded on a specially modified Brown Electronik Recorder. This equipment permitted measurement of head travel with an accuracy of 0.0001 in. Although the measurement included any deflection of the gripping system, comparative deflection measurements with Huggenberger extensometers on the gage section of a specimen at various loads indicated that the creep in the specimen was obtained with an accuracy of 1 per cent. To determine the creep strain in the specimen from the deflections, it was necessary to determine the effective gage length, taking into consideration the creep in the fillet section of the specimen. The effective gage length was calculated by assuming a hyperbolic sine creep function[3] and by using a graphical integration of strain over the straight section and fillet section of the specimen. This procedure was conducted for each specimen. The range of the effective gage lengths was between approximately 110 and 130 per cent of the actual machined gage length.

The stress-rupture tests were conducted in Baldwin-Southwark stress-rupture testing machines, 20,000-lb capacity, equipped with automatic creep-time recorder. For determination of creep strain from the recorded elongation, the effective gage lengths were determined as described above. The elevated temperature tension tests were conducted in a standard universal testing machine equipped with a furnace and temperature controls. Stress-strain curves were obtained using Baldwin-Southwark extension arm autographic extensometers mounted on the gage length of the specimens. At least three tension specimens were tested at each temperature and the results averaged.

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[3] H. Neuber, "Theory of Notch Stresses," The David W. Taylor Model Basin, Translation 74, United States Navy, November, 1945.

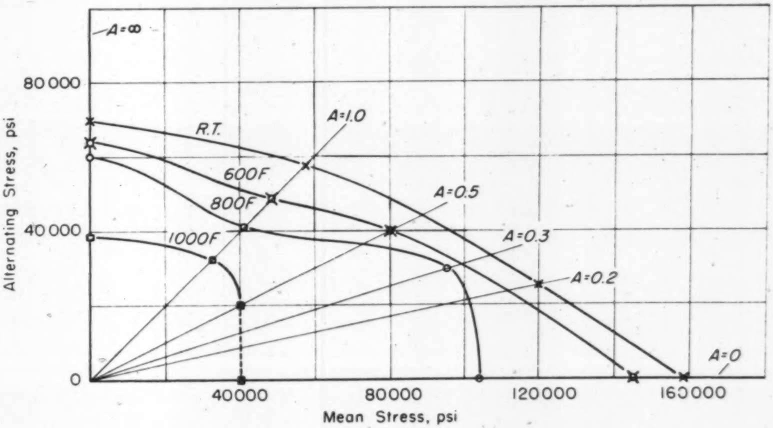

FIG. 3.—Alternating Stress-Mean Stress Diagrams at Various Temperatures for a Life of 15 × 10⁶ Cycles, Unnotched Specimens.

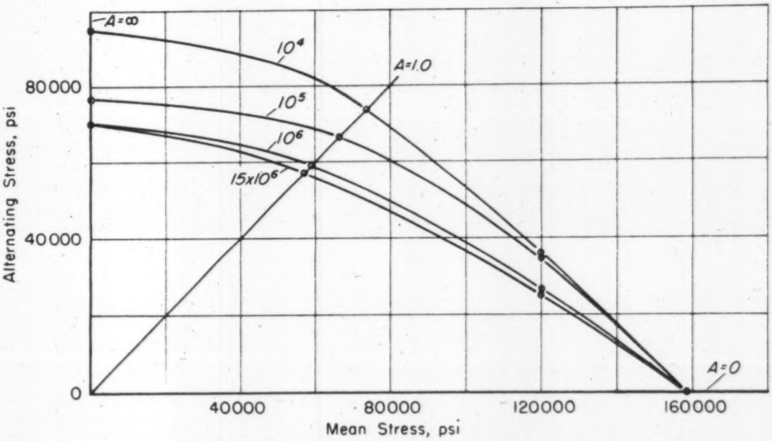

FIG. 4.—Alternating Stress-Mean Stress Diagrams for Different Lifetimes at Room Temperature, Unnotched Specimens.

RESULTS AND DISCUSSION

The tensile, 120 hr stress-rupture, and fatigue strengths at 15 × 10^6 cycles are given in Tables I and II for unnotched and notched specimens, respectively. It will be noted that a life of 15 × 10^6 cycles, in these tests, corresponds in time to 120 hr. The 120-hr stress-rupture values were interpolated or extrapolated from stress *versus* time to failure curves plotted from the actual test data given in Table III.

The fatigue test results are presented in the form of alternating mean stress diagrams for the different stress ratios and lifetimes from 10^4 to 15 × 10^6

cycles (Figs. 3 to 11). Throughout this paper, the stress ratio used is indicated as A, defined as the alternating stress divided by the mean stress. The stress ratios used were chosen so as to produce the most accurate alternating-mean stress diagrams possible with the limited number of specimens available. These diagrams were derived from the experimentally determined stress-cycle curves, one for each stress-ratio, temperature, and notched or unnotched condition. These stress-cycle curves are not given, due to lack of space. However, there is a point to be noted in connection with these elevated temperature stress-cycle

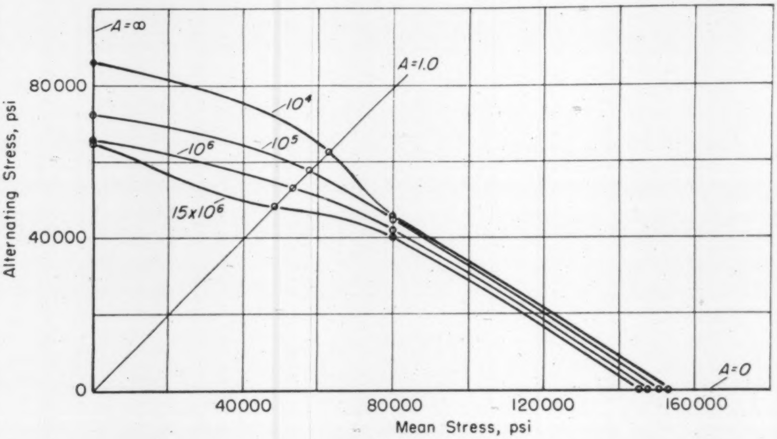

FIG. 5.—Alternating Stress-Mean Stress Diagrams for Different Lifetimes at 600 F, Unnotched Specimens.

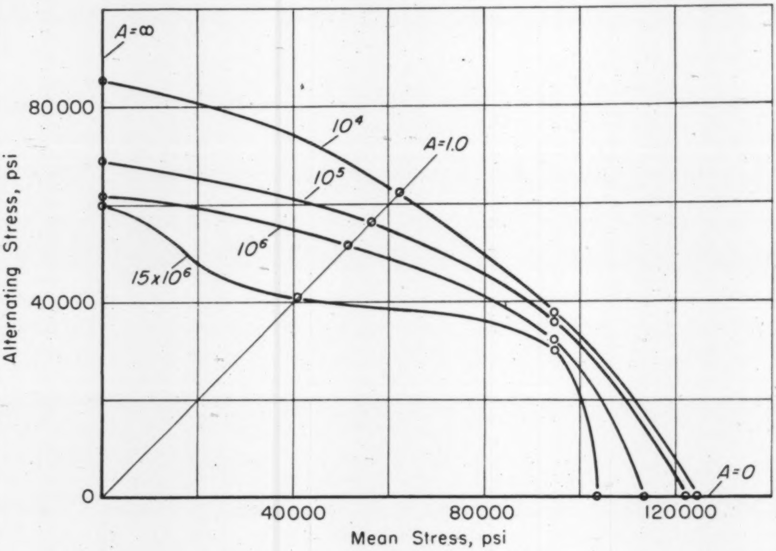

FIG. 6.—Alternating Stress-Mean Stress Diagrams for Different Lifetimes at 800 F, Unnotched Specimens.

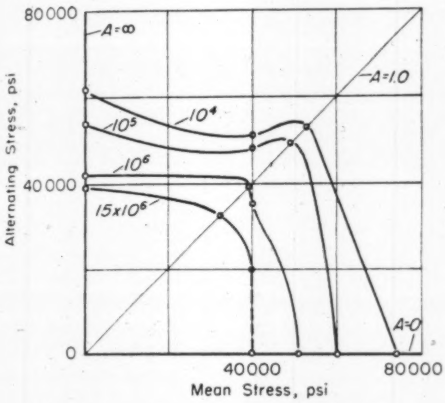

FIG. 7.—Alternating Stress - Mean Stress Diagrams for Different Lifetimes at 1000 F, Unnotched Specimens.

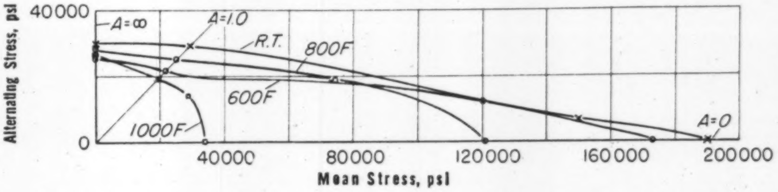

FIG. 8.—Alternating Stress-Mean Stress Diagrams at Various Temperatures, for a Life of 15 × 10^6 Cycles, Notched Specimens.

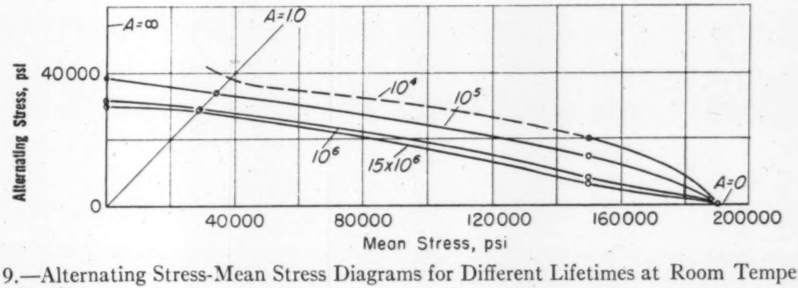

FIG. 9.—Alternating Stress-Mean Stress Diagrams for Different Lifetimes at Room Temperature, Notched Specimens.

curves. Although, for steel at room temperature, the fatigue strength at about 10^7 cycles is the endurance limit, it should not be assumed that the elevated temperature fatigue strengths at 15×10^6 cycles reported in this paper are endurance limits. Tests were not carried beyond this point, but for sufficiently high temperature and mean stress, the curve undoubtedly continues to drop, due to creep effects, and even for other elevated temperature conditions, it has not been established that these values are endurance limits.

In constructing the room temperature alternating-mean stress diagrams for zero ordinate, the ultimate tensile strength was plotted as the abscissa. However, at elevated temperatures, because of creep, stress-rupture strengths were plotted instead, for lifetimes equal to the corresponding fatigue curve, such as 120 hr for 15×10^6 cycles, and correspondingly shorter times for the curves for 10^6, 10^5, and 10^4 cycles. For the notched condition, the ultimate tensile strength and creep-rupture strengths for notched specimens were used for the points on the alternating-mean stress diagrams.

Nondimensional diagrams were constructed from the alternating-mean stress data by plotting $\frac{S_a}{S_e}$ as ordinate *versus* $\frac{S_m}{S_c}$ as abscissa. The 45-deg straight line from an ordinate of 1 and an abscissa of 0 to an abscissa of 1 and an ordinate of 0, is a modified Goodman straight-line relationship with the equation:

$$\frac{S_a}{S_e} = 1 - \frac{S_m}{S_c}$$

where S_a is allowable alternating stress for specified lifetime or number of cycles and for specified mean stress S_m; S_e is the experimental fatigue strength at the same life time for completely reversed stressing; and S_c is the experimental static stress-rupture strength, or tensile strength at room temperature, for specified lifetime converted to hours. All the fatigue data for the unnotched condition, for all lifetimes covered at all temperatures, are above this straight line. The data at 1000 F are the farthest above this line. For the notched condition, all

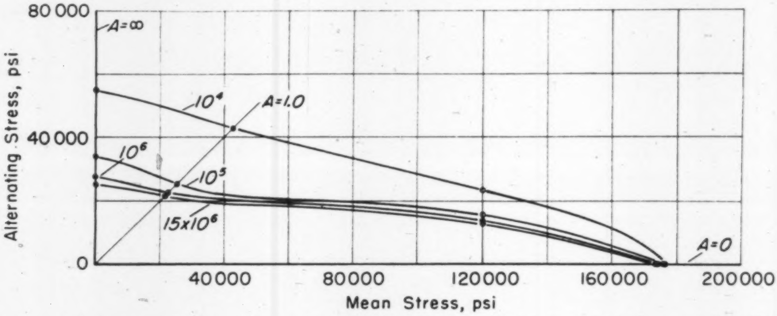

FIG. 10.—Alternating Stress - Mean Stress Diagrams for Different Lifetimes at 600 F, Notched Specimens.

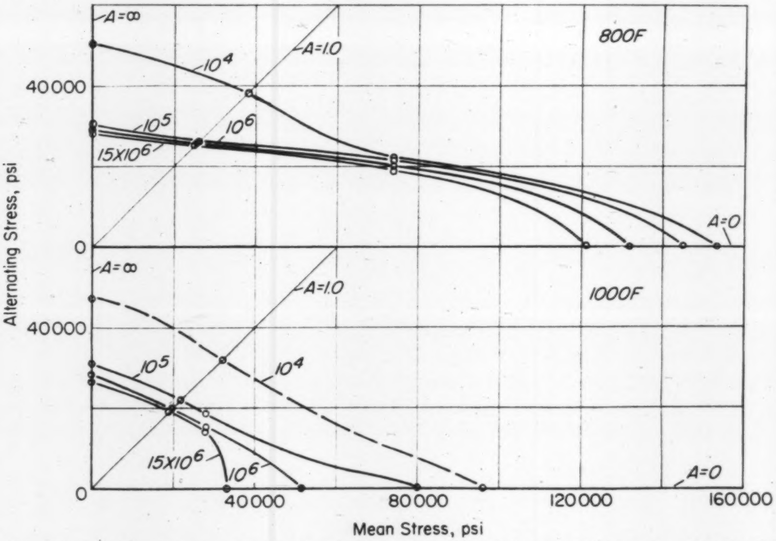

FIG. 11.—Alternating Stress - Mean Stress Diagrams for Different Lifetimes at 800 F and 1000 F Notched Specimens.

the data at all temperatures for 15 × 10^6 cycles are on or above this straight line, although they are closer to the line than for the unnotched condition. However, for shorter lifetimes (higher stress levels), the data for notched specimens fall below the line in some instances.

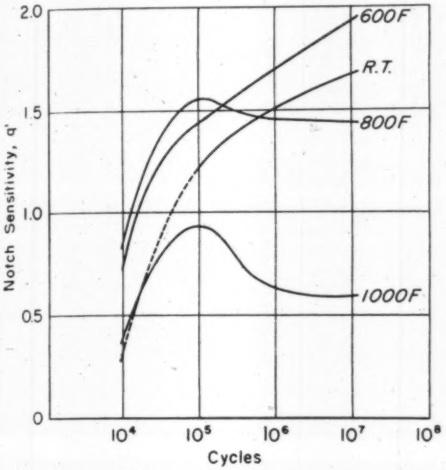

FIG. 12.—Fatigue Notch Sensitivity as Function of Life for Various Temperatures, Completely Reversed, $A = \infty$.

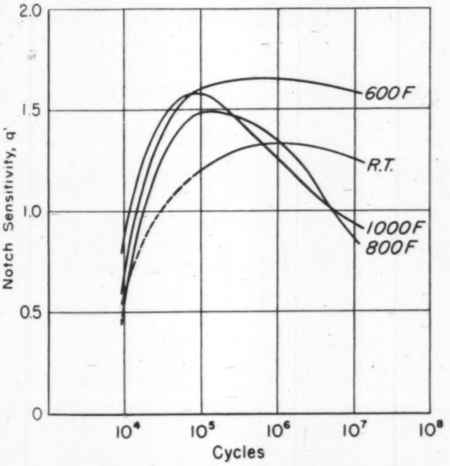

FIG. 13.—Fatigue Notch Sensitivity as Function of Life for Various Temperatures, Tension, Zero to Maximum, $A = 1$.

Fatigue notch sensitivity as a function of life, temperature, and stress ratio is shown in Figs. 12 to 14. The notch sensitivity index employed is defined as:

$$q' = \frac{K_f - 1}{K_{t'} - 1}$$

where K_f = fatigue strength reduction factor and $k_{t'}$ is the "technical" stress concentration factor, derived from H. Neuber's "Theory of Notch Stresses."[4] The factor q' was used, based on $K_{t'}$, rather than the factor q, based on K_t, because the value of $K_{t'}$, a corrected factor which takes into consideration the effect of the flank angle, comes closer to the actual fatigue reduction factors found in the investigation (see Table II) than K_t, which is the theoretical elastic stress concentration factor. For the notch used in this investigation (Fig. 1) $K_{t'} = 1.8$ and the corresponding theoretical stress concentration factor $K_t = 3.3$; therefore q is $q' \times 0.348$.

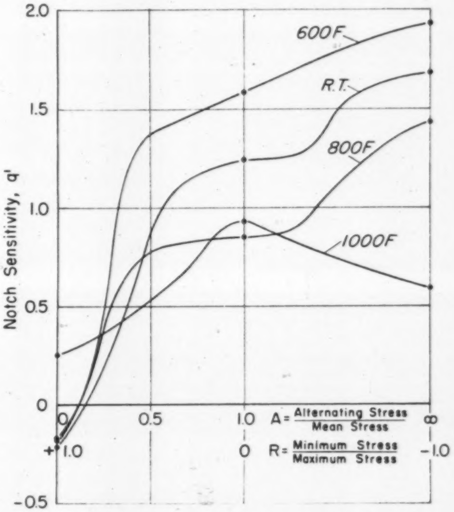

FIG. 14.—Fatigue Notch Sensitivity as Function of Stress Ratio for Various Temperatures at a Life of 15 × 10^6 Cycles.

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[4] A. Nadai, "The Influence of Time upon Creep. The Hyperbolic Sine Creep Law," Stephen Timoshenko Anniversary Volume, Macmillan Co., New York, N. Y., p. 155 (1938).

Figures 12 and 13 show the notch sensitivity, q', as function of life (or stress level) for the two different stress ratios $A = \infty$ and $A = 1$. For the stress ratio $A = \infty$, Fig. 12, the notch sensitivity was a maximum at 800 F and 1000 F at a stress level corresponding to a life or number of cycles between 10^5 and 10^6. However, at room temperature and 600 F the notch sensitivity continued to rise up to the maximum number of cycles tested. The highest values of notch sensitivity were at 600 F, except for the highest stress level corresponding to lives or numbers of cycles up to about 10^5 where the notch sensitivity was higher at 800 F. For the stress ratio $A = 1$, Fig. 13, a maximum appears to be reached for all temperatures, at from 10^5 to 10^6 cycles. The notch sensitivity is greatest at 600 F, except below 10^5 cycles, where the curve for 1000 F is highest. Figure 14 shows the influence of stress ratio on notch sensitivity at different temperatures at 15×10^6 cycles. The trend is decreasing notch sensitivity with decreasing stress ratio at all temperatures tested with the exception between $A = \infty$ and $A = 1$ at 1000 F. The peak of notch sensitivity was at stress ratio $A = \infty$ for all temperatures except for 1000 F.

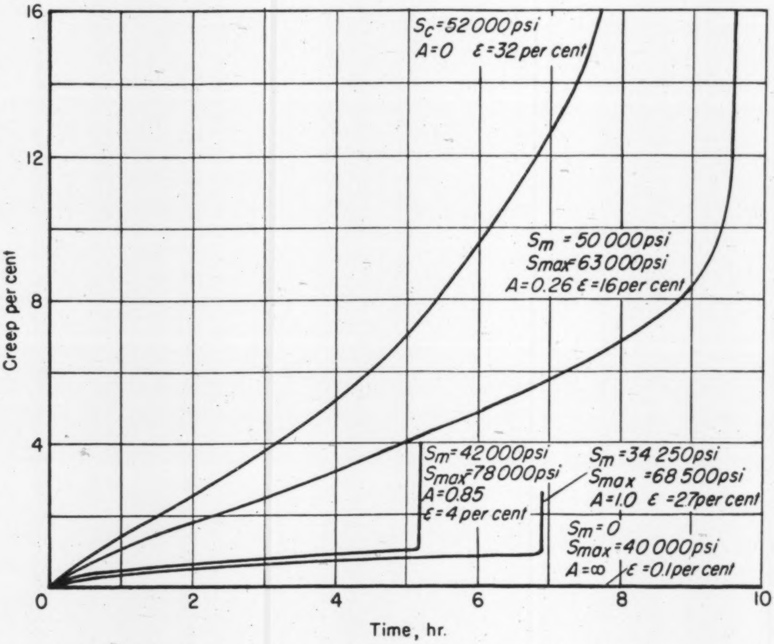

Fig. 15.—Creep-Time Diagrams at 1000 F, Unnotched Specimens.

In Figs. 15 and 16 a few creep curves are presented. The curves in Fig. 15 for various stress ratios all at 1000 F, show that creep is dependent on mean stress rather than maximum stress. At a sufficiently high mean stress, the type of creep curve changes from one that produces relatively little creep in the second stage and practically no third stage creep at all, to one that is typical of static creep time curves. In the former case, there is an abrupt change from second stage creep to the typical fatigue type failure. In the latter, a stress-rupture failure is obtained. At lower temperatures, this change in the type of creep curve does not occur until the stress is close to the yield strength. In static stress-rupture tests, unnotched specimens, it is noted from the data in Table III that for the temperatures below 1000 F, the rupture stresses are about equal to, or higher than, the short-time yield strength at these temperatures. The percentage strain values shown in Figs. 15 and 16 are the elongations of the failed specimens. The elongation is negligible for completely reversed stressing, even at 1000 F, is small for those cases where third stage creep is negligible, and is large when the creep curve is similar to the static condition.

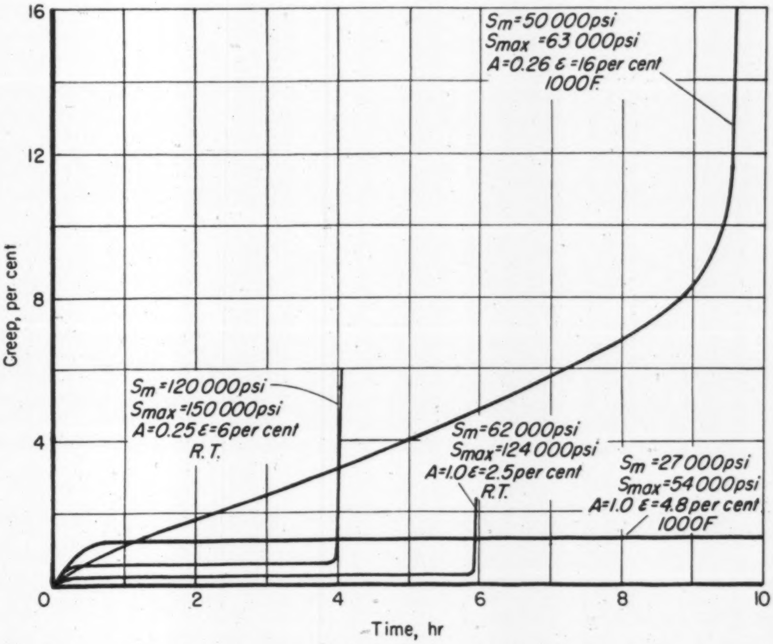

FIG. 16.—Creep-Time Diagrams, Unnotched Specimens.

The ratios, K_f, of the experimentally determined fatigue strength at 15×10^6 cycles of unnotched specimens to that of notched specimens are all well above 1.0. However, the comparable values for tensile strengths and 120-hr creep-rupture strengths are all below 1.0, except for stress-rupture strength at 1000 F. These factors below 1.0 indicate good ductility under these conditions. It will also be noted from Table III that the reduction of area values of notched stress-rupture specimens are lowest at 1000 F, corresponding to the conditions for the highest notch factor. This investigation was conducted on steel quenched and tempered to 160,000 psi tensile strength. The same steel, tempered to 208,000 and 125,000 psi tensile strengths, is now being investigated and will be reported on in subsequent papers.

SUMMARY AND CONCLUSIONS

Alternating mean stress diagrams for SAE 4340 steel of 160,000 psi tensile strength were presented for unnotched and notched specimens for 10^4 to 15×10^6 cycles at room temperature, 600, 800, and 1000 F. Tensile and stress-rupture properties on corresponding specimens at these temperatures were also obtained.

Within the temperature range investigated, the fatigue strengths for unnotched specimens decrease as the temperature increases at all stress levels and stress ratios. The fatigue notch sensitivity is dependent upon temperature, stress level and stress ratio, but no generally valid correlations were found. The temperature at which highest fatigue notch sensitivity was shown, was at 600 F. However, the stress-rupture notch sensitivity was greatest at 1000 F.

Creep is more dependent upon mean stress than upon maximum stress. The failure characteristics change from pure fatigue to pure creep as the stress ratio decreases from $A = \infty$ to $A = 0$ at elevated temperatures. Two distinct types of creep-time diagrams were obtained for high and low stress ratios.

At elevated temperatures appreciable creep occurs under dynamic loading at all stress ratios except $A = \infty$. At stress-ratio $A = \infty$ the ultimate failure is pure fatigue, but at all other stress ratios the ultimate failure may be fatigue or creep depending upon the relative magnitude of the effects of stress, temperature, and time.

Where creep is involved at elevated temperatures, fatigue life is dependent not only on the stress and number of cycles, but also on time. Therefore, the speed of loading (cyclic frequency) must be considered at elevated temperatures since it determines the length of time in which creep may occur.

DISCUSSION

MR. J. M. LESSELLS.[1]—This is an interesting paper since it deals with the effect of notch sensitivity in the blue brittle range. From work published by the Mond Nickel Co.[2] on special alloys it was found that under a combination of mean stress and a variable stress the maximum stress sustained by the material at different elevated temperatures was always in excess of the rupture strength for that particular temperature. The comparison was made on the basis of a similar time period for the fatigue and rupture tests. Comments by the authors on this aspect would be of interest.

MR. T. J. DOLAN.[3]—I should like to ask two questions of the authors. First, since the Schenck machine did not have a "load maintainer," I am wondering what method was used to readjust the machine to maintain the mean stresses shown in the alternating stress-mean stress diagram; or was the machine readjusted during the test at all?

Second, what value was plotted to obtain the abscissa on the alternating stress-mean stress diagrams at $A = 0$, that is, the mean stress value with no accompanying alternating stress. Was this the short-time tensile strength or a creep-rupture stress for some particular time? If so, does this time correspond with that involved in the fatigue test? It always raises the question as to just what is the significance of rupture strength at elevated temperature under steady stress and whether values should be based on the original cross-sectional area, the necked-down cross-sectional area, or whether it should be prescribed by some lifetime over which the test is to be significant?

MR. B. J. LAZAN.[4]—In computing K_t, what value of Neuber's ρ' was used? Was one of Neuber's constants used or was it otherwise determined?

MR. E. J. WARD[5] (*presented paper for authors*).—In answer to Mr. Lessells' question about the work of the Mond Nickel Co., this effect has been found in some cases of heat-resisting alloys under conditions of high mean stresses and low alternating stresses. I think Mr. Lazan has found that same condition in his work.

In answer to Mr. Dolan's first question about the load maintainer, the load was adjusted during the test. Of course, in most of the work there was no creep involved, that is, in all the lower temperatures, and the stress ratio $A = \infty$, so we did not have to worry there. However, under the conditions where creep did occur we periodically readjusted the static load by taking up on the screw protruding from the end of the machine which controls the static load.

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[1] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] Mond Nickel Co., "The Nimonic Series of Alloys—Their Application to Gas Turbine Design, London (1949).

[3] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[4] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.

[5] Chief, Fatigue Section, Structural Design Data Branch, Materials Laboratory, Directorate of Research, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

In regard to the second question as to which value we used in plotting our alternating stress-mean stress diagrams, at $A = 0$, we used the creep-rupture value at that temperature for 120 hr. We obtained that by testing several specimens in creep and interpolating or extrapolating as necessary, to get the stress for rupture in 120 hr. We felt this was the proper way to do it, and we chose 120 hr as that is the time it takes our machine to reach 15×10^6 cycles, which was used as the endurance limit in the tests.

In answer to Mr. Lazan's question, in using ρ' we did use Neuber's value there.

DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF UNNOTCHED AND NOTCHED N-155 ALLOY AT ROOM AND ELEVATED TEMPERATURES*

BY L. J. DEMER[1] AND B. J. LAZAN[1]

SYNOPSIS

Data are presented on the dynamic properties of N-155 alloy under rotating bending stress. Results of tests on unnotched and notched specimens at room temperature, 1350 F, and 1500 F are included. Existence of a cyclic stress sensitivity limit for damping at all three temperatures is shown. Damping of notched specimens is discussed in relation to the volume of material effective in dissipating resonant vibration energy. Dynamic stress-strain data for unnotched specimens and stress-deflection data for notched specimens are presented. Changes in vertical deflection and runout for the notched specimens are associated with the first evidence of the formation of a fatigue crack. Curves for number of cycles to first evidence of crack plotted against stress are compared with the usual *S-N* fracture diagrams. Fatigue strength reduction factors are determined from both the fracture and first-evidence-of-crack data. The latter values are found to be nearly independent of the number of fatigue cycles. Use of the damping of notched specimens as a possible criterion for the notch sensitivity of materials in fatigue is discussed.

Interest in the dynamic properties of materials stems from two distinctly different points of view. The rapid pace of development of new propulsion engines and other similar applications requires a better understanding of the properties of the component materials under severe dynamic conditions and over wide temperature ranges. Furthermore, the desire for a more thorough understanding of the fundamental properties of materials demands knowledge of the changes occurring in the damping and elasticity properties during continued alternating stressing and of their relationship to notch sensitivity, fatigue damage, crack propagation, and ultimate fracture.

General relationships existing among damping, elasticity, and fatigue in design considerations have been discussed previously (**1**).[2] More recent work (**2**) attempts to provide a method for predicting the behavior of a member under resonant conditions from the material's dynamic properties together with the cross-sectional and longitudinal stress conditions on the part. In another study (**3**), an analytical method for determining the internal damping of machine members from property data and design-loading information is offered. There is considerable

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Research Fellow and Professor of Materials Engineering, respectively, University of Minnesota, Minneapolis, Minn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 851.

interest in the comparative significance of low and high stress damping and the relative contribution of each to the limitation of destructive amplitudes at resonance. Before this matter can be resolved for all applications, the damping behavior of the materials over a wide range of stress is necessary.

A knowledge of the damping and stiffness behavior of materials is needed to further the basic understanding of fatigue failure. For this purpose, interest is centered in the intermediate and high ranges of stress from perhaps 50 to 150 per cent of the fatigue strength. As an extension of interest, at least for certain classes of materials, it is necessary to include temperature as a variable.

This paper is concerned with dynamic properties of the temperature-resistant alloy N-155 which has been used in jet engine turbine applications. The test program included rotating-bending fatigue tests on both unnotched and notched specimens performed at room temperature, 1350 F, and 1500 F. The work was undertaken as a continuation of an earlier study of similar materials (1), and to supplement and correlate with a NACA cooperative study (4) on the relationship between the static and fatigue properties of this alloy.

TABLE I.—SUMMARY OF TEST DATA.

	Room Temperature	1350 F	1500 F
UNNOTCHED SPECIMENS			?
Static proportional limit,[a] psi	40 500	22 900	20 300
Static yield strength[a] 0.2 per cent offset, psi	60 500	36 900	35 800
Static tensile strength,[a] psi	119 000	60 200	44 600
Elongation in 2 in.,[a] per cent	44.0	26.7	22.5
Static modulus of elasticity,[b] psi	30.0×10^6	22.2×10^6	21.0×10^6
Fatigue strength at 2×10^7 cycles, psi	53 000	40 000	29 000
Ratio of FS to static tensile strength	0.445	0.665	0.650
Stress at cyclic stress sensitivity limit, psi	34 000	26 000	25 000
Ratio of stress at CSSL to FS at 2×10^7 cycles	0.64	0.65	0.86
Specific damping at CSSL, in-lb per in.^{3} per cycle	0.55	2.3	3.0
Slope of D-S line below CSSL	3.0	3.0	3.0
Slope of D-S line above CSSL	8.6–10.0	4.3–9.6	8.4–8.6
Specific damping at FS at 2×10^7 cycles, in-lb per in.^{3} per cycle	30–40	15–140	10–12
Approx. dynamic proportional limit, psi	40 000	34 000	29 000
Ratio of DPL to FS at 2×10^7 cycles	0.76	0.85	1.00
Ratio of DPL to S at CSSL	1.18	1.30	1.16
NOTCHED SPECIMENS			
Fatigue strength at 2×10^7 cycles, psi	24 000	21 500	19 500
Fatigue Strength Reduction Factor, K_f', at 2×10^7 cycles	2.2	1.85	1.5
Ratio of notched FS to unnotched TS	0.20	0.36	0.44
Slope of D_o-S line	3.6	3.6	3.6
Total damping, D_o, at FS at 2×10^7 cycles, in-lb per cycle	0.0007	0.002	0.002

[a] Average of values reported by University of Michigan (4).
[b] Average of results from tension and reflectoscope tests, University of Michigan.

MATERIAL AND TEST METHOD

The low-carbon N-155 alloy investigated was a portion of the lot of material procured by the NACA for use in a previously reported program (4). Details on the chemical composition and stock processing schedule may be found elsewhere (4). The static physical properties are given in Table I.

The unnotched type specimen used was shown previously (1). The notched type specimen employed in these tests has a notch of 0.206-in. minimum diameter with a 0.010-in. root radius and straight sides with 60-deg included angle. The notch is in the center of a $2\frac{1}{2}$-in. cylindrical length of 0.55-in. diameter.

The theoretical stress concentration factor, K_t, in bending is 2.6 as determined by the method of Neuber (**8**).

Information on the preparation of the specimens may be found elsewhere for the unnotched (**4**) and notched (**5**) specimens. The testing machine and details of the test method have been described in earlier papers (**1**,**6**).

RESULTS AND DISCUSSION

Fatigue Properties:

Fatigue test results for both specimen types at the three test temperatures are plotted in Fig. 1. Since only a small number of points were obtained for each curve, the diagrams presented are only approximate. Two curves are shown for the notched specimens at each temperature. The solid lines indicate the number of cycles to fracture of the specimen. The dashed lines indicate the number of cycles to the first evidence of fatigue cracking, as obtained from changes in the damping and stiffness behavior of the specimens.

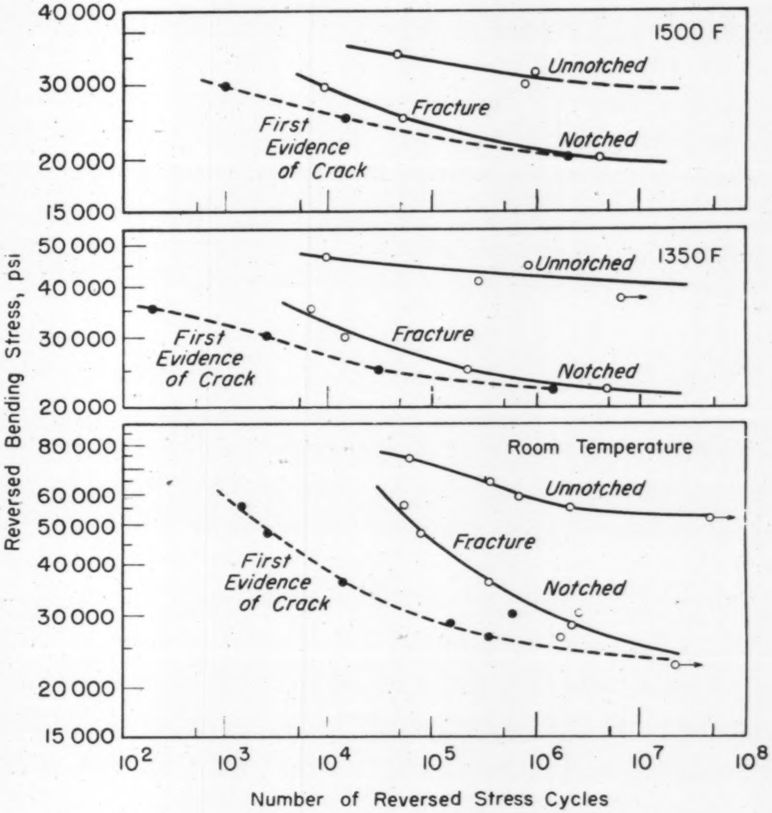

FIG. 1.—*S-N* Fatigue Fracture Curves for Unnotched and Notched Specimens and First-Evidence-of-Crack Curves for Notched Specimens of N-155 at Room Temperature, 1350 F, and 1500 F.

The approximate fatigue strength values at 2 × 10^7 cycles, obtained from the curves of Fig. 1, are given in Table I. These indicate lower unnotched and notched fatigue strengths with increasing temperature. However, an accompanying decrease in the fatigue strength reduction factor, K_f, with temperature, for example, 2.2 at room temperature, 1.85 at 1350 F, and 1.5 at 1500 F, indicates a decrease in notch sensitivity.

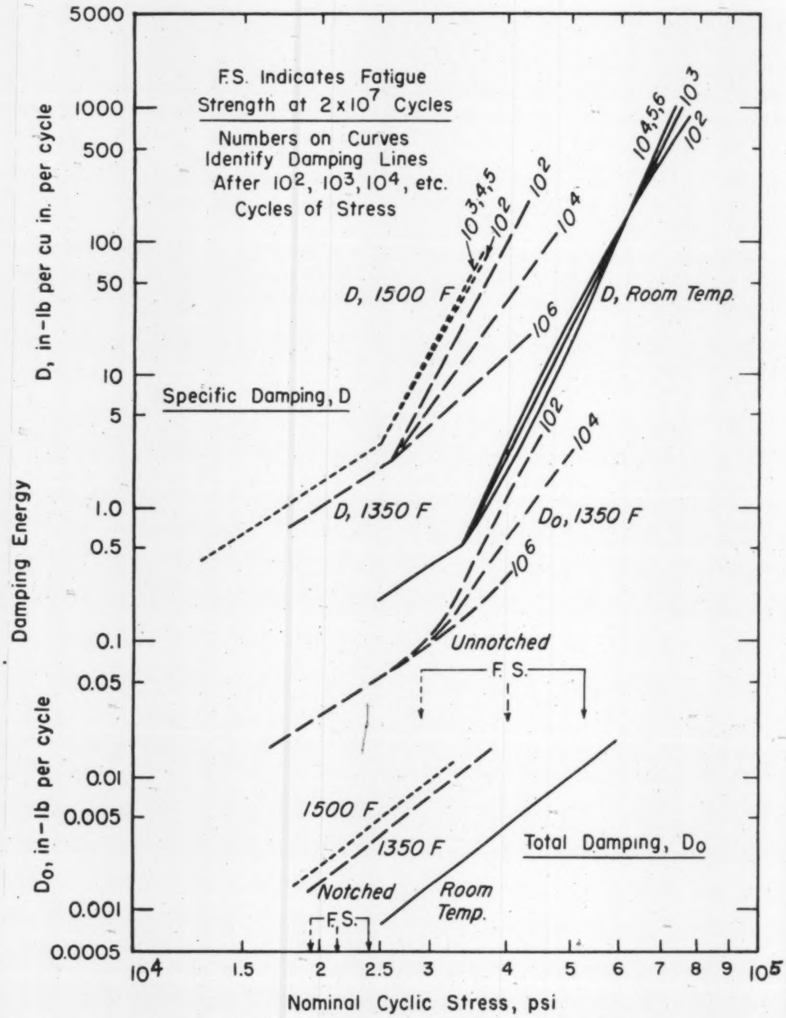

FIG. 2.—Effect of Stress Magnitude and Constant Cyclic Stress History on Damping Energy for Unnotched and Notched N-155 at Room Temperature, 1350 F., and 1500 F.

It was impossible to determine the first evidence of cracking from the damping or deflection data of the unnotched specimens. Since no other suitable method was available at the time for detecting the early stages of cracking in these specimens, first-evidence-of-crack curves are not presented for this type.

Damping Properties:

During the fatigue tests, microscope readings were taken of the horizontal displacement (upon reversal of rotation) of a target in the end of the cantilever loading arm attached to the specimen. These readings were continued at intervals to the fracture of the specimen.

Using formulas presented in Appendix I of another publication (5), the displacement readings were converted to values of total damping energy in in-lb per cycle dissipated by the entire specimen. The total damping is the energy absorbed per cycle by the total volume of stressed material. It is not a basic unit since the total damping for a given maximum stress is dependent on the stress distribution in, and size of, the specimen. At all three temperatures, the damping of the unnotched specimens is independent of stress history in the lower stress ranges. Above certain stress values, however, a stress history effect is apparent.

In the case of each notched specimen, the damping during the early part of the fatigue test was unaffected by the number of cycles. Later, at a point significantly before fracture, abrupt changes in damping occurred which can be associated with the appearance of macrocracks in the specimen and a significant decrease in specimen stiffness.

A summary of the damping data obtained is presented in Fig. 2. In the lower portion of the figure, the total damping energy, in-lb per cycle, for the notched specimens is plotted as a function of the nominal stress at the notch roots. There is no effect due to stress history indicated, since the damping values prior to the first evidence of cracking were constant and only these values were considered. The slope values for the notched curves on the log-log plot, approximately 3.6, are independent of temperature and display no change in the stress range covered.

All of the fatigue strengths obtained are indicated in Fig. 2 for comparison purposes. The notched specimens, at the two elevated temperatures, show the same energy dissipation at their respective fatigue strengths. These values are more than twice the corresponding room temperature value.

The lowest branched curves in Fig. 2 show the total damping energy for the unnotched specimens tested at 1350 F. The stress history effect is observed above a stress of approximately 26,000 psi. Beyond this point each curve represents the damping values obtained after a given number, 10^2, 10^4, etc., of applied stress cycles.

Specific damping energy, D, may be calculated from the total damping values, D_0, by means of the relationships given in Appendix I of (5). As used here, specific damping energy is the energy absorbed per cubic inch of material under conditions of uniform direct stress. It is the damping energy associated with a given unit stress and is expressed in in-lb per cu in. per cycle. Values of specific damping for the unnotched specimens at the three test temperatures are shown in the upper portion of Fig. 2.

Comparing the total and specific damping curves for 1350 F, it is seen that the conversion to specific damping for the unnotched specimens results in a rather abrupt change in slope at a certain value of stress, rather than the more gradual curvature shown by the curves for total damping. Furthermore, the point of change in slope appears to be the stress at which the effect of stress history begins to be manifested. This point has been called (7) the "cyclic stress sensitivity limit," abbreviated CSSL. Such a limit has been observed with most of the various materials so far investigated. The stress and damping values at the CSSL, and the location of the CSSL in per cent of the fatigue strength are given in Table I for the three temperatures.

Considering the stress range investigated, the straight lines on the log-log plot for specific damping *versus* stress indicate that up to a certain stress value (for this material, the CSSL) specific damping is related to stress by the equation:

$$D = JS^n$$

where:

J and n = constants for a given temperature,

and S = the maximum alternating stress in psi.

Beyond this critical stress, different values of J and slope, n, are required to specify the curves corresponding to different temperatures and stress histories. The values of n and J, as determined from the individual curves of Fig. 2, are given in another publication (**5**). Below the CSSL, the curves for unnotched specimens have a slope of 3.0. Above the knee, the slope values fall in the range from approximately 4 to 10. These large values of the exponent are of particular interest and significance. It has been considered (**9**) that, at engineering stress levels, materials generally display an exponent of 3. In the case of N-155, this is true only up to the CSSL, but above that value, as with most of the other heat-resistant materials investigated to date, the n values are considerably larger than 3. It is apparent that extrapolation of low stress damping measurements to higher stresses of engineering importance may be grossly misleading. Similarly, with certain materials, deviations from linearity of the log D *versus* log S relationship may occur at lower stresses than the range considered here (**10**).

The small total damping energy, D_0, absorbed by the notched specimens, as shown in Fig. 2, may be extremely important when resonant members are considered. Comparisons made between the damping of the unnotched and notched specimens, at corresponding temperatures and considering stress concentration factors, indicate an extremely small effective volume of the notched specimen as far as damping is concerned. For one such comparison using the room temperature data, an effective volume for the notched specimens in the order of 0.000006 cu in. is obtained. The term "effective volume" is taken as that volume of material under uniform direct stress which would dissipate an equivalent amount of energy. Notches act as serious stress raisers under the application of fatigue stresses. Furthermore, they should be avoided in members expected to contribute significant material damping against resonant vibration.

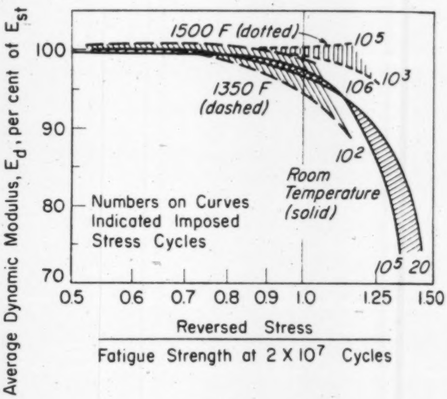

FIG. 3.—Effect of Stress Magnitude and Stress History on Average Dynamic Secant Modulus of Elasticity for Unnotched N-155 Specimens at the Three Test Temperatures.

Elasticity Properties:

Records were kept of the vertical deflection of the unnotched specimens during the course of each fatigue test. These data were converted to values of average dynamic modulus of elasticity (a secant value) by the relationships given in Appendix I of (**5**). The term "average" is used to denote that these values were obtained from deflection readings for a beam in bending rather than under uniform direct stress conditions. They are not the values associated with a given stress, but are representative of the bending stress distribution existing over the whole cross-section. A value of 30.0 ×

10^6 psi was used for the room temperature static value.

Although there is a considerable decrease in the dynamic modulus of elasticity for the room temperature specimens at the higher stresses, the decrease for a given ratio of stress to fatigue strength is comparable to that at 1350 F, as Fig. 3 indicates. The average dynamic modulus in per cent of static modulus is plotted on the basis of stress ratio. The data for 1500 F show the smallest change in modulus with stress ratio of the three test temperatures. The maximum decrease in dynamic modulus due to stress history only is about 6 per cent at the 1350 F fatigue strength. The maximum decrease observed due to both stress history and stress magnitude is about 25 per cent for the highest test stress at

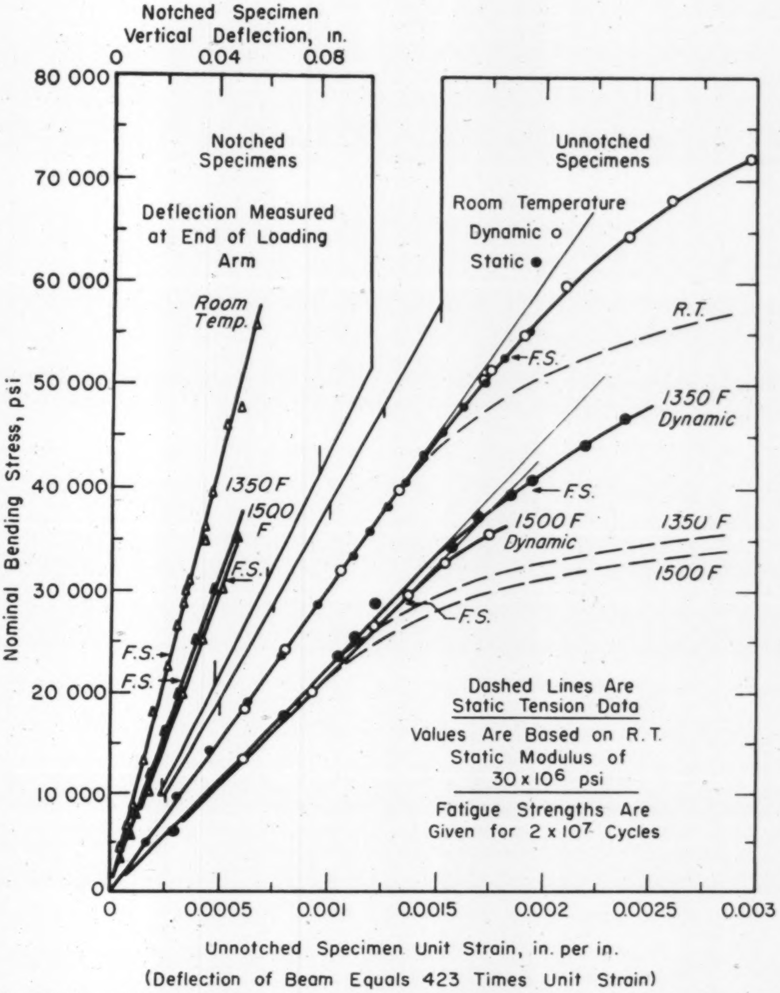

FIG. 4.—Static and Dynamic Stress-Strain Curves for Unnotched and Dynamic Stress-Deflection Curves for Notched N-155 Specimens.

room temperature. The effects of large changes in dynamic modulus on resonant frequency and amplitude of vibration have been discussed previously (**1**).

Dynamic Stress-Strain Curves:

Dynamic stress-strain and stress-deflection curves for the unnotched and notched specimens, respectively, are shown in Fig. 4 along with static tensile data. Each point for the dynamic data represents a different test made, in most cases, on a different specimen. The dynamic stress-strain relationship shown is that after 100 cycles of reversed bending stress. In this figure, the large circles represent the dynamic test data, the small solid points are data from a static room temperature bending test on a single specimen, and the dashed curves represent the static tension data obtained by Michigan (**4**).

At all three temperatures the static tensile proportional limit falls below the dynamic bending proportional limit. The static bending curve at room temperature, however, practically coincides with the dynamic bending curve. At 1500 F the fatigue strength of N-155 is very close to the dynamic proportional limit, whereas at the other two temperatures it is at a stress about 20 per cent above the dynamic proportional limit. The values determined as the proportional limits, however, are dependent on the sensitivity of the testing equipment, as well as on small differences in specimen effective gage lengths due to machining tolerances. These factors could account for scatter in the curves which might shift the dynamic proportional limit by 10 per cent or more.

The dynamic load-deflection curves for notched specimens, shown in Fig. 4, indicate linearity even in the high stress regions well above the fatigue strengths. The points for these curves were taken after 100 cycles of stress before any evidence of fatigue cracking was apparent. On the basis of the changes in dynamic modulus (in Fig. 3), it might be expected that a gradual decrease in the slope of the stress-deflection curves would be exhibited at the higher stress levels. However, as discussed previously in connection with the damping data, the effective volume of metal at peak stress in the notched specimens is extremely small, apparently too small to affect significantly the total deflection of the specimen attributable to a change in modulus of elasticity.

First-Evidence-of-Crack Curves for Notched Specimens:

Measurements of vertical deflection[3] and runout at the target recorded during the course of each notched fatigue test are an aid in determining the start of macro-cracks. These data are plotted in Fig. 5 for the room temperature tests. It is observed that there are significant changes in both deflection and runout at points between 3 to 15 per cent of the fracture life of a specimen. These are marked ΔV and ΔR. At high fatigue stresses the ΔV points are the best indication of cracking. At lower stresses both the ΔV and ΔR points serve as an indication. This is the result of the manner of crack propagation in specimens of high stress concentration discussed by Peterson (**11**).

Interpretation of the deflection, runout, and damping curves for each notched specimen was made to obtain the points plotted on the dashed curves of Fig. 1 labeled the "first-evidence-of-crack" curves. It is estimated that uniform crack depths of less than 0.0005 in. may be detected by this method. A number of checks were made on the actual presence of fatigue cracks both before and after the detection of a change in deflection during the notched tests at room temperature. An optical microscope was used. No cracks were detected prior to the increase in deflection, whereas they were always observable after the increase. Crack searches of this type were not undertaken for the elevated temperature tests due to experimental difficulties. However, the deflection curves obtained for the high temperature tests were similar in all details to those of Fig. 5.

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[3] The deflection is determined at the measuring target which is approximately 17 in. from the free end of the cantilever-beam specimen. See References (**1**) and (**2**) for schematic diagrams and photographs of the testing machine.

As previously pointed out, there was no pronounced increase in vertical deflection of the unnotched N-155 specimens which could be associated with the first evidence of a fatigue crack until immediately before failure. Work is now in progress on methods for detecting the initiation of cracking in such specimens. All the comparisons which follow between the unnotched and notched specimens refer to the S-N fracture curve for the unnotched specimens. Is it assumed that if first-evidence-of-crack curves were available for the unnotched specimens, they would fall so close to the fracture curves as not to affect appreciably the relationships now to be considered.

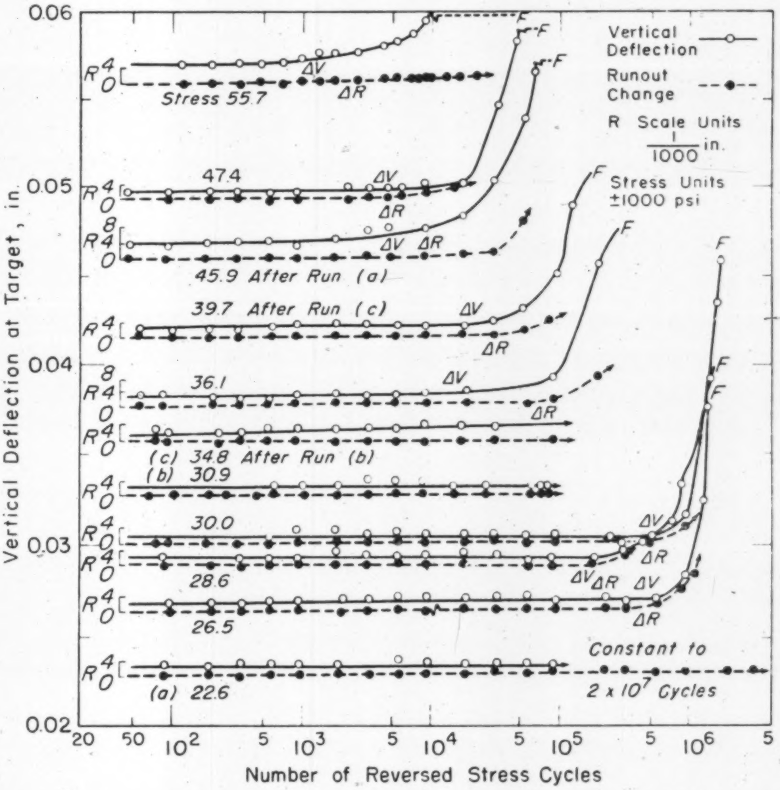

FIG. 5.—Effect of Sustained Cyclic Stress of Various Magnitudes on the Vertical Deflection and Runout of Notched N-155 Specimens at Room Temperature.

Initiation and Propagation of Fatigue Cracks:

Examination of Fig. 1 discloses that at all three test temperatures the first-evidence-of-crack curve is considerably flatter than the fracture curve for the notched specimens; also, the spread at a given nominal stress between the two curves is greatest for room temperature. Variation in methods of plotting the information contained in the graphs of Fig. 1 affords a number of interesting relationships. In this manner indications may be obtained regarding both the initiation of the fatigue cracks in the notched specimens and the rate of propagation of the cracks.

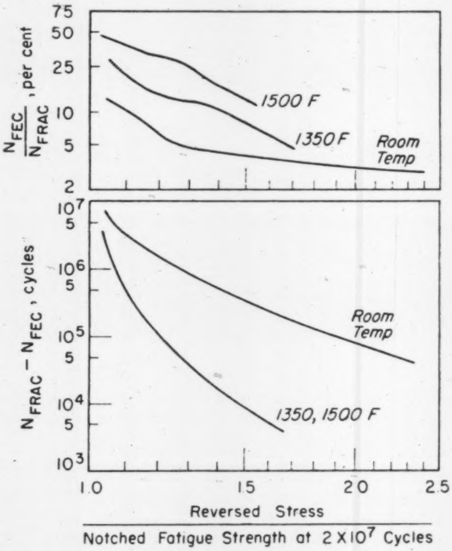

FIG. 6.—Per cent of Fracture Life at Which First Evidence of Crack Appears and Number of Cycles Between FEC and FRAC for Notched N-155 at the Three Test Temperatures.

Figure 6 shows that on the basis of the same ratio of test stress to fatigue strength the first evidence of cracking occurs at a lower percentage of total cycles to fracture as the test temperature is lowered. Plotting the number of cycles between the first-evidence-of-crack curves and fracture curves for the notched specimens at the various temperatures as a function of ratio of test stress to fatigue strength indicates a smaller number of cycles as the test temperature is raised. It is shown by Fig. 6 that the relative rate of crack growth increases with increasing temperature. Since the notch sensitivity of this material decreases with increasing temperature, as shown in Table I, the rapid crack growth may be due to the increased rate of oxidation which takes place at elevated temperatures.

Notch Sensitivity:

Referring to Fig. 1 it may be observed that the ratio of the notched fracture strength to the unnotched fracture strength increases with increasing stress; that is, the two fracture curves for each temperature tend to converge in the short life region. Conversely, the first-evidence-of-crack curves in each case remain much more nearly parallel to the fracture curves of the unnotched specimens in the life ranges investigated. To interpret and analyze this behavior, the definition of the fatigue reduction factor, or effective stress concentration factor, should be reviewed.

The usual fatigue strength reduction factor, based on the ratio of the fracture stress for the unnotched specimen to the fracture stress for the notched specimen at a given number of cycles, is given the symbol K_f, whereas K_f' shall be defined here as the ratio of the fracture stress for the unnotched specimen to the stress at the first evidence of cracking, at the same number of cycles, for the notched specimens. When both K_f and K_f' are plotted as a function of number of stress cycles, as shown in Fig. 7, the K_f' curves are reasonably independent of the life of the specimen, but the K_f values increase with number of cycles. It should be mentioned here that if the K_f' values were computed from first-evidence-of-crack curves for unnotched as well as notched specimens, they would be even more nearly constant throughout the specimen life. The possibility that K_f' may provide a more significant indication of the effect of stress concentration in fatigue than does K_f is now being investigated for other materials. The theoretical stress concentration factor for the notched specimens according to Neuber (8) is indicated in Fig. 7 for comparison purposes.

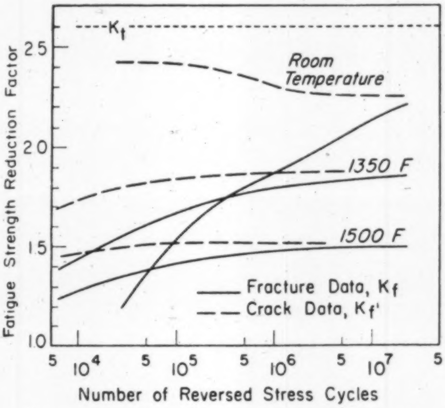

FIG. 7.—Fatigue Strength Reduction Factors as a Function of Stress Cycles Showing K_f Values from Fracture Data and K_f' Values Determined from First-Evidence-of-Crack Data for N-155.

In an investigation (not yet reported) of the dynamic properties of various materials at room temperature, a possible correlation was observed between the damping of notched specimens and the notch sensitivity of the materials. A commonly used measure of notch sensitivity is the index:

$$q = \frac{K_f - 1}{K_t - 1}$$

If a new index is defined as:

$$q_o = \frac{K_{f'} - 1}{K_t - 1}$$

with K_f' based on the first-evidence-of-crack curve rather than on the fracture curve, then the index q_o is substantially independent of the number of cycles. Values computed in this manner for q_o at the three test temperatures, for 10^6 cycles of stress, are approximately 0.81, 0.55, and 0.32, respectively, for the room-temperature, 1350-F, and 1500-F test conditions. The values of q_o may vary between zero and one, with the larger value indicating greater notch sensitivity.

A possible association between the fatigue notch sensitivity and the damping of notched specimens of N-155 is noted in the observation that, upon comparing the total damping for the three test temperatures at equal stress, see Fig. 2, the greatest energy dissipation occurs at 1500 F, for which temperature the notch sensitivity index, q_o, has the smallest value. In partial contradiction to this, when compared at their respective fatigue strengths, the data for the two elevated temperatures show equal energy dissipation even though q_o has a different value for each temperature, see Table I. It appears from other work currently in progress, however, that a measure of the notch sensitivity in fatigue for various materials, at the same temperature, might be given by the relative damping properties of notched specimens compared on the basis of equal ratio of test stress to fatigue strength with the notch sensitivity being inversely proportional to the damping energy.

SUMMARY AND CONCLUSIONS

The approximate unnotched fatigue strength values obtained at room temperature, 1350 F, and 1500 F, are in good agreement with those obtained by other investigators (4). The notched fatigue strength values indicate a decreasing fatigue strength reduction factor as the temperature is raised.

The shapes of the specific damping *versus* stress curves are generally similar at all three test temperatures. Those for the unnotched specimens plotted on a log-log basis show a linear variation from the lowest stresses investigated (about 45 per cent of the fatigue strength) up to the cyclic stress sensitivity limit (CSSL). In this range where stress history has no effect on damping the slope of the linear curves on the log-log plot is approximately 3.0 for all three temperatures. Beyond the CSSL, the damping curves at all three temperatures are again approximately linear on the log specific damping *versus* log stress plot but with steeper slopes ranging from approximately 4 to 10.

The damping data for the notched specimens satisfy straight lines on the log total damping *versus* log stress plot throughout the stress range from approximately 1.0 to 2.5 times the notched fatigue strength at each temperature. The curves at all temperatures have approximately the same slope, 3.6. A comparison of the damping in notched and unnotched specimens indicates that an extremely small volume of material in a notched specimen is effective in dissipating resonant vibration energy.

For the unnotched specimens at the three test temperatures, the decrease in average dynamic secant modulus of elasticity due to both stress and stress history is approximately 6 per cent at the fatigue strength in the most variant case of 1350 F. There is, however, a pronounced decrease in stiffness at both room temperature and 1350 F for stress values considerably in excess of the fatigue strength. The dynamic load-deflection curves for notched specimens are linear even in the high stress regions well above the fatigue strengths.

First-evidence-of-crack curves for the notched specimens are considerably flatter than the fracture curves. At the higher test stresses, the first evidence of cracking occurs at a lower percentage of the total specimen fracture life than at lower stresses. Cracking was detected as early as 3 per cent of the fracture life in room temperature notched specimens. At stresses near the long time fatigue strength, the crack and fracture curves tend to approach each other although, due to the use of a log scale for N, a small difference represents millions of cycles.

Even though the material became less notch sensitive with increasing temperatures, curves showing the number of cycles between the first evidence of cracking and fracture in the notched specimens indicate higher rates of crack propagation at the elevated temperatures.

Values of q_o, an index of notch sensitivity determined by using fatigue strength reduction factors for crack data (rather than fracture data), are substantially independent of the number of cycles. Comparison of the damping results for the notched specimens at a given stress for the three test temperatures shows that the highest damping is associated with the lowest notch sensitivity.

Acknowledgment:

The help of D. Patterson, J. Wachenheim, and J. Winslow, of the University of Minnesota, in the procurement of data is gratefully acknowledged. The fine cooperation of the U. S. Air Force, sponsors of this work, is appreciated. The equipment used in this investigation was developed under both Office of Naval Research and U. S. Air Force sponsorship.

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(2) B. J. Lazan, "Effect of Damping Constants and Stress Distribution on the Resonance Response of Members," *Journal of Applied Mechanics*, Vol. 20, p. 201 (1953).

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(7) B. J. Lazan and T. Wu, "Damping, Fatigue, and Dynamic Stress-Strain Properties of Mild Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 639 (1951).

(8) H. Neuber, "Theory of Notch Stresses," J. W. Edwards, Ann Arbor, Michigan (1946); The David W. Taylor Model Basin, *Translation No. 74*, Nov., 1945.

(9) J. M. Robertson and A. J. Yorgiadis, "Internal Friction in Metals," *Journal of Applied Mechanics* Vol. 13, No. 3, p. A173 (1946).

(10) A. W. Cochardt, "The Origin of Damping in High-Strength Ferromagnetic Alloys," Paper 52-A-21 contributed by the Applied Mechanics Division for presentation at the Annual Meeting of the Am. Soc. Mechanical Engrs., New York, N. Y., Nov. 30–Dec. 5, 1952.

(11) R. E. Peterson, Interpretation of Service Fractures, Chap. 12, "Handbook of Experimental Stress Analysis," John Wiley and Sons, Inc., New York, N. Y (1950).

DISCUSSION

Mr. P. R. Toolin.[1]—The authors note that for notched specimens the rate of crack propagation apparently increases with increasing test temperature, despite a concomitant decrease in notch sensitivity. What would be the results for a similar comparison of unnotched specimens? Were any of the specimens which "ran out" sectioned and examined? We sectioned notched specimens which had not failed after more than one hundred million cycles at a temperature of 1200 F and have found very small fatigue cracks at the notch base. These cracks either started very late or progressed very slowly.

With specimens tested in repeated bending at room temperature, we have also noted that fatigue cracks, in some materials, may start very early in the life of the specimen. It has also been observed that sometimes there is a large difference between the number of cycles to the appearance of a minute fatigue crack and the number of cycles to the appearance of the crack which progresses to failure. I wonder if the authors have observed this in their investigation.

In regard to the fact that the notch sensitivity index of N-155 was found to decrease as the test temperature was increased, it should be noted that N-155 is a very stable alloy and that this trend may not apply to all alloys. Thus for a wrought alloy of less stability than N-155, we have found the notch sensitivity index, q, to rise from 0.4 at room temperature to 0.6 at 1200 F, and then to decrease to 0.25 at 1500 F.

Mr. Fred Keller.[2]—Some results we obtained are different from these shown here in that when we plotted first slip and then first crack we did not get a decrease in the number of cycles between the first crack and the first slip as was indicated on the authors' curves.

Actually what we are doing with this method is detecting much finer cracks than Mr. Lazan.

The first cracks occur within a grain as fissures along crystographic planes. These cracks are extremely fine and would not be picked up by ordinary microscopic examination. The next stage in the progress of such cracks is that they join; many people have seen this type of fatigue crack. Cracks usually start in grains of certain orientation and then progress from one grain to another.

In the preliminary work that we have done along this line with these methods, following the surface of the specimen practically every cycle if we wish, we have obtained some evidence of cracking after only a few at certain stresses. We can determine first deformation, first cracking along the crystographic planes, joining of cracks, and finally when the cracks progress from grain to grain.

Preliminary indications are that it may be possible to use this method to determine the probable life and endurance limit of the material by plotting either first slip or first crack.

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[1] Mechanics Department, Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] Chief, Metallography Division, Aluminum Company of America, New Kensington, Pa.

MR. J. M. LESSELLS.[3]—The work reported by the authors is of extreme interest since, to have an understanding of the inner mysteries of the fatigue problem, this sort of approach will have to be made.

It is interesting to note that the authors were able to detect the first crack by a study of the sidewise deflection. This approach was mentioned, I believe, first by Inglis.[4] In this regard, Mr. Lazan was fortunate in the choice of the rotating bar machine which allows such deflection studies to be made. This approach would be denied in the case of a direct stress type.

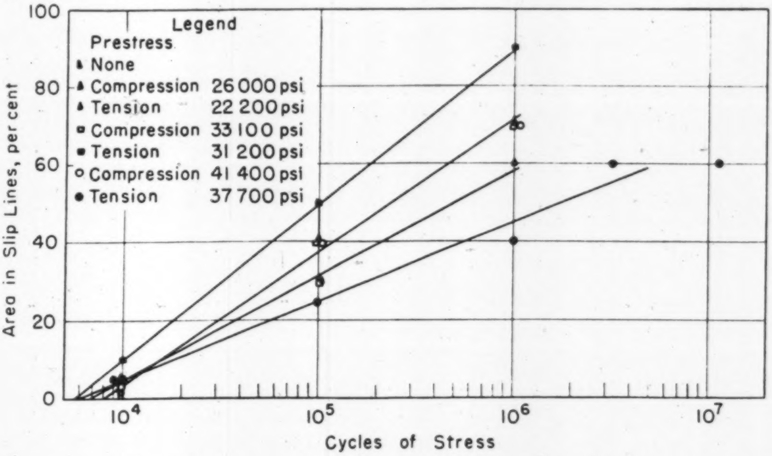

FIG. 8.—Area in Slip Lines at 30,000 psi Fatigue Stress.

We do know that elastic hysteresis changes with increase in stress even though the stress level is within a safe range. This study of hysteresis may throw some light on the behavior prior to the appearance of a crack.

From my own work[5] in cyclic bending on crack development it was necessary to destroy the specimen to ascertain if a crack had been found. This procedure was also adopted by Phillips,[6] using cyclic direct stress. A further study of the sidewise deflection may lead to useful results.

MR. T. J. DOLAN.[7]—Mr. Lazan and his co-workers are to be congratulated on an excellent piece of work in trying to study basically what is involved in the initiation of fracture. Certainly it is a complicated subject and, as has been pointed out, fatigue damage occurs at a very early stage in the fatigue process.

In this connection the accompanying Fig. 8 shows some unpublished results obtained at Illinois.[8] These data were obtained by studying with the aid of the light microscope the slip in the crystalline structure on the surface of a small beam and plotting the per cent of the surface area involved in slip. If one extrapolates these back to zero ordinate (for no slip) they indicate a number of cycles varying from 5000 to 8000 for first visible evidence of damage. Presumably it would be difficult by this method of observation to detect slip in the early stages. However, there is damage by slip starting early and progressing with a fairly linear relationship with regard to the logarithm on the number of cycles until fracture occurs.

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[3] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] N. P. Inglis, "Hysteresis in Metals Under Alternating Stresses," *The Metallurgist*, Vol. III, Sept. 30, 1927, p. 138.

[5] J. M. Lessells and H. E. Jacques, "Effect of Fatigue on Transition Temperature of Steel," *Welding Research Supplement*, No. 2, pp. 74–83 (1950).

[6] C. E. Phillipps and R. B. Heywood, "The Size Effect in Fatigue of Plain and Notched Specimens Loaded Under Reversed Direct Stress," *Proceedings*, Inst. Mech. Engrs., Vol. 165 (W.E.P. No. 65), pp. 113–124 (1951).

[7] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[8] W. J. Love, "Structural Changes in Ingot Iron Caused by Plastic and Repeated Stressing," ONR Project NR-031-005, *Tech. Report No. 33*, Dept. Theoretical and Applied Mechanics, University of Illinois, Nov., 1952.

By examining specimens with the electron microscope periodically, very microscopic cracks that could not be seen with light microscope were detected at a very early stage (as low as 0.1 per cent of the fatigue life to fracture).

The conclusion that the fatigue strength reduction factor was independent of life agrees with observations made in a recent study of brass.[9] For each of six groups of the metal each having a different grain size, we found that the fatigue strength reduction factor was practically constant from a few thousand cycles up to 20 million cycles.

To revert to the authors' discussion of damping, it was noted in some of Lazan's earlier work that rest periods and frequency of stressing seemed to affect the damping exhibited by the specimens. I would like to ask whether the frequency of stressing or the presence of rest periods would have an effect on the occurrence of the "stress sensitivity limit" defined in this paper. If a member is loaded to a stress level beyond the stress sensitivity limit, will rest periods or variations in frequency of stressing markedly affect the damping?

MR. B. J. LAZAN (*author's closure*).—Most of the questions raised by the discussors deal with the initiation and propagation of cracks. Since the primary aim in this work was to determine the damping, elasticity, and fatigue properties, the test program was designed primarily from this viewpoint, rather than to procure conclusive answers to many of the cracking problems of current interest. Therefore, some of the questions raised cannot be answered conclusively at this time.

The authors assume that Mr. Toolin was referring to unnotched specimens in the second paragraph of his discussion. No specific attempt was made to study the early cracking of unnotched specimens of N-155. In other materials, however, pre-failure cracking in unnotched specimens has been observed and in many cases is very pronounced.

Mr. Keller has done some fine work in the development of techniques for detection of very early slipping and cracking. This type of approach should aid considerably in defining the mechanism of early fatigue damage. However, the applicability of this method to notched specimen work, particularly at high temperature, requires considerable further development, and this should be encouraged. If also large surface areas are involved (authors' test specimen has 1.3 sq in.) a very difficult searching job is required.

It should be mentioned that the crack data presented in this paper are by no means intended to indicate the point of crack initiation in the notched specimen. Much more elaborate technique and equipment than that employed in this investigation would be required for detecting the formation of cracks within the grains. The data presented are concerned with what is referred to by some investigators as the third stage of fatigue, that is, crack growth which ultimately leads to fracture. It is believed that the first-evidence-of-crack line represents an early stage in the development of the crack causing failure.

In regard to cracking and fracture it is interesting to inquire whether the first-evidence-of-crack curve joins or lies below the fracture curve in the long-life region. Evidence by Lessells and Jacques (1950)[5] and Fenner and Fwen (1951)[10] among others, indicate that at least for some materials, the curve for crack initiation tends to remain below the fracture curve. This would indicate that there are small fatigue cracks which grow so slowly that for most practical purposes stabilized conditions may be assumed. More information on this point is desirable but, needless to say, extremely difficult to obtain because of the large scatter and long-time problems involved in obtaining statistically sound data.

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[9] R. W. Karry and T. J. Dolan, "Influence of Grain Size on Fatigue Notch-Sensitivity," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 789 (1953).

Mr. Lessells' comment regarding the method employed for detection of early cracks should be clarified. A change in vertical deflection method was used rather than a change in horizontal traversal. In regard to the sensitivity of this method, it should be explained that the deflection is read by means of an optical micrometer microscope at a target on the end of a cantilever beam. The target has a motion approximately 1000 times the unit strain in the specimen. If the crack were around the full periphery of the notch, a crack of some 0.0002 or 0.0003 in. depth could be detected. However, if it were just at one point on the periphery of the specimen, its depth would have to be considerably greater for detection. Even though this method is more convenient, it is not as positive as the destructive method of sectioning used by Lessells.

Mr. Dolan's approach to the cracking problem, like Mr. Keller's, provides a very sensitive indication of the stages preceding the authors' "first-evidence-of-crack" joints. The very early cracking indicated by Dolan's data should again be noted.

With regard to Mr. Dolan's inquiry on the effect of frequency on the damping, it should be mentioned that all damping data reported herein were procured at 20 cpm. Frequency has been observed to be important in the higher damping range, usually above the cyclic stress sensitivity limit, where frequency effects may cause a 10:1 change in damping in some materials. The frequency effect for N-155 material was not determined since this added variable would greatly enlarge the test program.

Although in these tests damping was measured at 20 rpm, higher frequency was used between measurements to shorten the fatigue tests. In earlier work (7)[11] it was found that an increase of frequency between readings did not appear to affect the damping *versus* number of cycles curve.

With regard to the effect of rest periods, it was observed in prior work on mild steel (7) that rest may affect damping at stress levels above the cyclic stress sensitivity limit. Rest up to a few hours at zero stress after a period of cyclic stressing at a given stress level does not seriously affect the damping and modulus when measured again at the given stress. For longer rest periods the damping tends to decrease with time at rest.

The authors wish to thank the discussors for their aid in clarifying some of the problems brought up by this work.

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[10] A. J. Fenner, N. B. Owen, and C. E. Phillips, "The Fatigue Crack as a Stress Raiser," *Engineering*, Vol. 171, May, 1951, p. 637.

[11] See list of references appended to the paper, p. 851.

STRENGTH PROPERTIES OF ROLLED ALUMINUM ALLOYS UNDER VARIOUS COMBINATIONS OF ALTERNATING AND MEAN AXIAL FATIGUE STRESSES*

By B. J. Lazan[1] and A. A. Blatherwick[1]

Synopsis

Fatigue data on 14S-T6, 24S-T4, and 75S-T6 aluminum alloys tested under various combinations of mean and alternating axial stress are presented. Stress range diagrams are included to analyze the effects of: (*a*) stress ratios in the range from static tension to reversed axial stress, (*b*) stress magnitude which causes failure in the range from 10^4 to 10^7 cycles, and (*c*) severity of circumferential notches having four different theoretical stress concentration factors in the range between 1.0 and 3.4. The extreme flatness of the stress range diagrams for severely notched specimens at long life is discussed in terms of the large reduction in mean load carrying capacity resulting from the addition of relatively small alternating stress. Unitless stress range diagrams are presented to indicate how material, life, and specimen type affect the combinations of alternating and mean stress which cause failure in a specified number of cycles. Data on the reduction in fatigue strength caused by notches are diagrammed to clarify the significance of mean stress, alternating stress, stress ratio, and cycles to failure as factors in fatigue notch sensitivity.

The inadequacy of fatigue data for structural aluminum alloys under various stress ranges has necessitated the use of rather broad assumptions in the design of aircraft structures. The strength data procured in this program at various stress ratios from static tension to reversed stress fatigue should help to alleviate this situation.

The strength potentials of aircraft materials are frequently not fully realized because of stress concentration effects. The notches generally present in aircraft structures necessitate, in the absence of adequate data and a basic understanding, a rather cautious approach and the inefficiency of overdesign is frequently the result. Notch sensitivity data procured in this work extend the range of stress ratios and fatigue lives covered in previous studies and provide improved prospective for structural designers.

Much of the prior work on fatigue properties of notched and unnotched aluminum alloys has been under reversed bending conditions (**1**–**5**)[2] in which the stress ratio $R = -1$, that is, the ratio of alternating to mean stress is infinite. The trend during recent years has been towards increased emphasis on axial load fatigue tests and consideration of other stress ratios. Although there have been several investigations on the unnotched and notched fatigue strength of aluminum alloys (**2**, **3**, **6**), it was not until very recently (**7**, **8**, **9**) that a reasonably wide range of stress ratios and notch effects were covered.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Professor of Materials Engineering and Director of Engineering Experiment Station, and Instructor, respectively, University of Minnesota, Minneapolis, Minn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 869.

TEST PROGRAM

All tests were conducted at room temperature under axial (tensile or compressive) stress. Stress magnitudes which caused failure in from 10^4 to 2×10^7 cycles, and in some cases to 10^8 cycles, were studied.

In order to cover as uniformly as possible the stress ratios ranging from static tension to reversed stress fatigue, selected steps of alternating-mean ratios (10) were used rather than selected steps of mean stress. The alternating-mean ratios A originally selected for this work were $A = 0$ (static tension), 0.37, 0.89, 2.16, and ∞ (reversed stress). As the project progressed, it was observed that in many cases small magnitudes of alternating stress seriously decreased the allowable mean stress, and therefore in such cases additional data were procured at alternating-mean ratios A of 0.15 and 0.08.

Materials:

The three rolled aluminum alloys used in this program were 14S-T6, 24S-T4, and 75S-T6. Further details on the material and its processing are given elsewhere (**18**).

The metallographic structure of the three test materials was examined by means of photomicrographs. Although the structures appeared to be reasonably normal, rather large insoluble constituents, which were probably chromium-bearing segregation, were found in the 75S-T6. Since the fatigue properties of this batch of 75S-T6 were lower than average, the Materials Laboratory of Wright Air Development Center and the Research Laboratories of the Aluminum Company of America both examined the material and reported (**11, 16**) the presence of chromium-bearing constituents larger than usually found in such rod. However, tests conducted by the Douglas Aircraft Co. (**17**) indicated that chromium-bearing segregation of the type observed does not necessarily cause low fatigue strength.

Specimens:

The four types of specimens used to obtain a range of stress concentration are described below. All specimens have a diameter of 0.400 in. at the test section. The unnotched specimen has a single fillet which is large enough to justify the assumption of zero concentration of stress (theoretical stress concentration factor $K_t = 1$). The mild-notch specimen has a semicircular circumferential notch of radius 0.10 in. and a theoretical stress concentration factor, as calculated from Neuber's charts (**12**), of 1.6. The intermediate-notch specimen has a circumferential 60-deg V notch with a 0.032-in. root radius resulting in a theoretical stress concentration factor of 2.4. The sharp-notch specimen has a circumferential 60-deg V notch with a 0.010-in. root radius and a theoretical stress concentration factor of 3.4.

Further details on specimen shape and method of preparation are given in reference (**18**). Since the introduction of internal stresses may seriously affect the fatigue properties, particularly of notched specimens, an attempt was made to determine the effect on fatigue properties of stress relief through a thermal treatment. Comparative tests showed that the stress-relief treatment had no significant effect.

Equipment:

Two different types of fatigue-testing machines were used to cover the range of axial stress required. An axial stress fatigue machine of the type described by Lazan (**10**) was used for low and medium alternating force tests, whereas a Sonntag fatigue machine with an amplifying fixture was used for high alternating force tests. Overlapping tests were made so that data from the two machines could be compared directly; no significant difference in fatigue properties was observed. Special axial stress grips were constructed for this program, using the approach described in an earlier paper (19).

RESULTS AND DISCUSSION

Static Tensile and Hardness Properties of Alloys:

In order to evaluate the uniformity of the three materials and also to determine how closely their properties compare with accepted values, a series of tension and hardness tests were undertaken. The tension tests were performed in accordance with ASTM standards (0.505 in. specimen diameter by 2-in. gage length) using a Baldwin universal testing machine and autographic recorder and the hardness tests were of the Rockwell A scale type. Data procured from specimens cut from the center and two ends of each of the 20-ft bars used are listed in Table I. The results of these tests agree closely with published data on these materials (2).

TABLE I.—STATIC PROPERTIES OF ALLOYS.

Material	Number of Tests	Hardness Rockwell A		Modulus E, 10^6 psi		Tensile Strength, psi		Yield Strength, 0.2 per cent Offset, psi		Elongation in 2 in., per cent	
		Average	σ[a]	Average	σ	Average	σ	Average	σ	Average	σ
14S-T6..........	21	51.3	0.60	10.5	0.15	71 600	400	63 500	480	13.6	0.84
24S-T4..........	21	48.5	0.75	10.6	0.12	72 800	550	48 600	480	21.4	0.61
75S-T6..........	21	56.0	0.50	10.4	0.10	82 300	390	70 900	540	16.5	0.50

[a] σ = standard deviation.

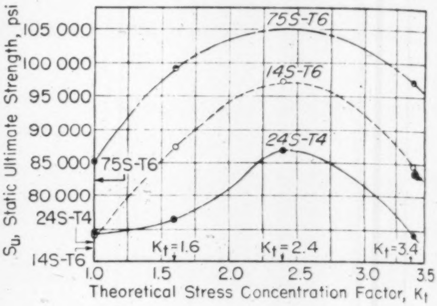

FIG. 1.—Variation of Static Ultimate Strength with Type of Specimen.

Static tension tests were also undertaken on the unnotched and notched type fatigue specimens used in this program. This was done not only to establish the general effects of the various types of notches on the static tensile strength but also to determine the zero stress ratio points for the stress range fatigue diagrams to be presented later. The results of these tests are diagramed in Fig. 1, the K_t values for all of the fatigue specimens being indicated along the abscissa. Also shown in this figure are the average tensile strengths for the standard straight-sectioned ASTM test specimen. Figure 1 shows that the tensile strength increases with increasing notch severity, reaching maximum at some intermediate K_t value, beyond which it decreases. Although such factors as stress gradient, plasticity properties, and the state of of stress (whether uniaxial, biaxial, or triaxial) are important, this effect is partially explainable on the basis that a fillet or notch has two major effects: (*a*) it causes stress concentration which tends to reduce the strength of the specimen, and (*b*) it restricts the reduction in area ("necking-down" prior to failure) which tends to increase the strength. Figure 1 shows that the strength of the single fillet unnotched specimen is larger in all cases than the ASTM specimen, and this may be explainable on the basis that factor (*a*) above is practically the same for both specimens whereas factor (*b*) favors the single fillet specimen. The peaked strength curve may similarly be explainable on the basis that factor (*b*) predominates up to a certain notch severity beyond which factor (*a*) becomes the more critical.

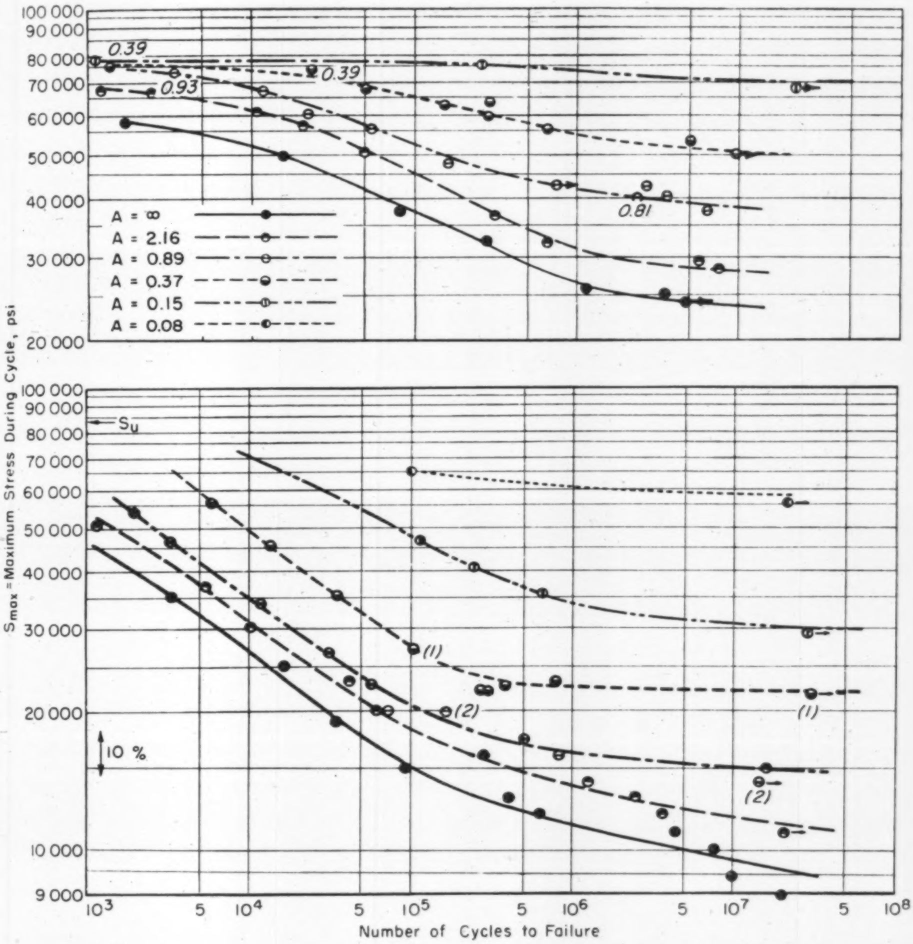

FIG. 2.—*S-N* Fatigue Diagrams at Various Stress Ratios for Unnotched and Notched Specimens of Aluminum Alloy 14S-T6.

Fatigue Properties of the Alloys:

The fatigue data procured on unnotched and sharp-notch specimens of 14S-T6 are plotted in Fig. 2 as *S-N* fatigue diagrams of the logarithm of the maximum or crest stress S_{max} during the cycle *versus* the logarithm of the number of cycles to failure. The crest stress S_{max} is used in these plots instead of the alternating stress S_a or mean stress S_m in order to improve the curve separation and clarity. Each alternating-mean ratio *A* is plotted as a separate *S-N* curve in

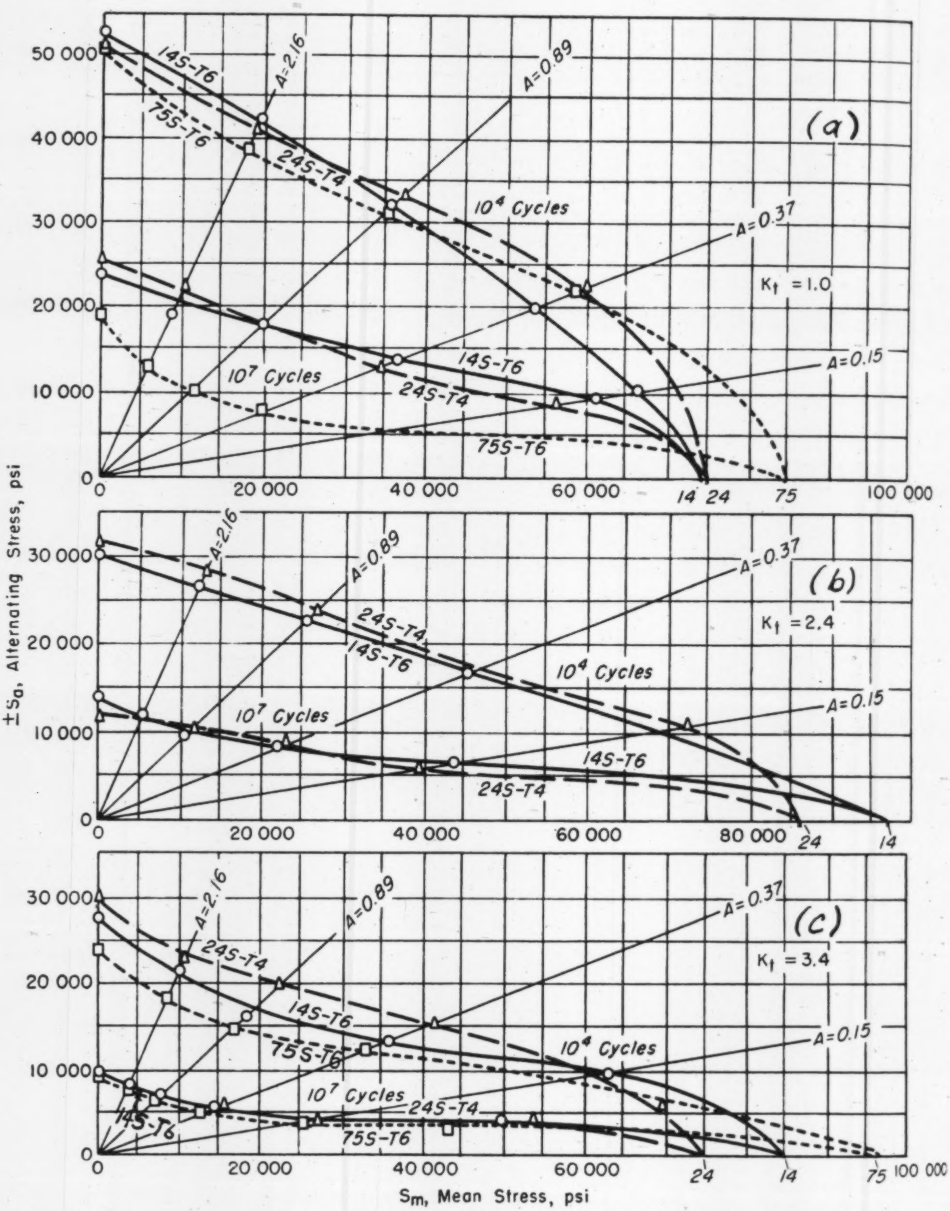

FIG. 3.—Stress Range Diagrams Showing Comparative Fatigue Strengths of Aluminum Alloys 14S-T6, 24S-T4, and 75S-T-6.

accordance with the point and line code shown.

Similar curves were plotted for the mild-notch and intermediate-notch specimens, but these are not included in this paper.

In general, it may be observed that the lower the alternating-mean ratio the smaller the slope of the *S-N* curve. The curve for $A = 0$ is not shown, but tests (18) indicated that the static stress-rupture data yield practically a horizontal line.

Since the scatter in fatigue strength data at long life is generally larger than at short life, it is rather difficult to establish the existence or non-existence of a horizontal asymptote or fatigue limit. Although the *S-N* curves presented in these figures and in other diagrams not included in this paper do not appear to approach the asymptote up to 10^7 cycles, nothing conclusive can be said on this matter.

Similar *S-N* diagrams were also plotted for 24S-T4 and 75S-T6 but are not included in this paper. The diagrams for 24S-T4 were very similar in all respects to those of 14S-T6, and the fatigue strengths were nearly identical. The curves for 75S-T6 were also similar in shape but indicated lower fatigue strengths, especially in the case of the unnotched specimens.

A comparison of the fatigue strengths of the three materials is given in Fig. 3. In these diagrams, the combinations of alternating and mean stress resulting in a given life are plotted in a coordinate system of alternating *versus* mean stress. The curves shown are the loci of these combinations for a fatigue life of 10^4 cycles and of 10^7 cycles for each material. The radial lines through the origin have a slope equal to the alternating-mean ratio used in the test program. The curves of Fig. 3(*a*) are for unnotched specimens while Figs. 3(*b*) and (*c*) are for notched specimens, $K_t = 2.4$ and 3.4 respectively.

No data were procured on 75S-T6 using the $K_t = 2.4$ notched specimens.

Comparing Figs. 3(*a*), (*b*), and (*c*), it is apparent that the higher the stress concentration factor in the specimen the flatter are the stress range curves, particularly for long life. Also of significance is the observation that although the curves for the unnotched and mildly notched specimens are generally either concave downward or straight, the severely notched specimens particularly at long life display curves which are concave upward. As a result of these trends, relatively small alternating force may greatly reduce the allowable mean stress. For example, referring to the curve in Fig. 3(*c*) for 14S-T6 and for a life of 10^7 cycles, the addition of an alternating stress of only 8 per cent decreases the allowable mean stress by 35 per cent from 84,000 to 54,000 psi, and an alternating stress of 15 per cent of the preload reduces the allowable mean stress 68 per cent from 84,000 to 27,000 psi.

Figure 3 also indicates the comparative fatigue strengths of 14S-T6, 24S-T4, and 75S-T6. Figure 3(*a*) shows that at the two lifetimes, the fatigue strengths for 14S-T6 and 24S-T4 are almost the same. The fatigue strength of 75S-T6, however, is much lower, especially at 10^7 cycles. The curves for 75S-T6 are much flatter in the low alternating-mean ratio region, for long life, a fact which emphasizes again the serious effect that a small vibratory stress may have on the allowable mean strength of this material.

In Fig. 3(*b*), constant-life curves for 14S-T6 and 24S-T4 are diagramed for specimens with an intermediate notch ($K_t = 2.4$). No specimens of this type were tested for 75S-T6. At 10^4 cycles, the fatigue strength of 14S-T6 is slightly lower than that of 24S-T4. However, at 10^7 cycles the strengths are almost identical. Figure 3(*c*) shows the fatigue strengths of the three materials for the sharp-notch ($K_t = 3.4$) specimens. Here the strength of 24S-T4 is the highest with 14S-T6 and 75S-T6 following in that order. At 10^7 cycles, the fatigue strengths of all three materials are practically the same.

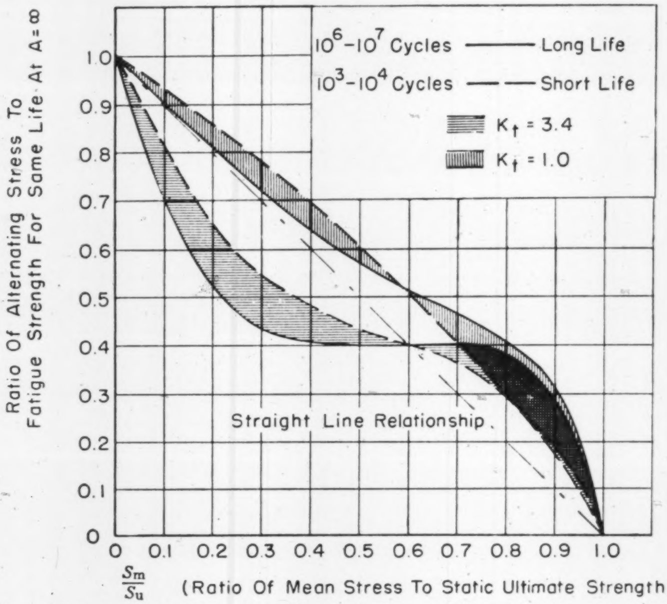

FIG. 4.—Unitless Stress Range Curves for Unnotched and Sharp Notched Specimens of 14S-T6 Aluminum Alloy.

It is desirable to determine if the stress range relationship is sufficiently consistent for all alternating-mean ratios to permit interpolation when only the reversed stress fatigue strengths and static strengths are known. In order to establish such a relationship, the stress range data for 14S-T6 are replotted on a completely unitless basis in Fig. 4. In this unitless diagram both the maximum ordinate and maximum abscissa are arbitrarily made equal to one. This means that the ordinate scale is the ratio of the alternating stress for a given life to the alternating stress for the same life under reversed stress conditions ($A = \infty$). The curves thus obtained fall within bands for each specimen type. Only the bands for the unnotched and the sharp-notched specimens are shown in Fig. 4.

The band for the unnotched specimens falls closer to the straight line relationship than does that of the notched specimens and it is above the line for all alternating-mean ratios. The band for the notched specimen is S-shaped, being below the line in the region of relatively high alternating stress and above the line for low alternating stress. The bands for the other two specimen types (not shown here) fall between these two extremes and display a gradual transition.

It may be noted that the long-life and short-life curves for both specimen types cross each other at a mean stress corresponding to six tenths of the static ultimate stress. This behavior was not found for either 24S-T4 or 75S-T6, however, though the general shape of the bands was otherwise quite similar, except that the 75S-T6 bands were generally below the straight-line relationship.

Notch Sensitivity:

It is apparent from the study of the foregoing stress-range diagrams that the reduction in fatigue strength caused by a notch is a function of not only dimensions but also stress ratios and stress level or life. The relationship of the notch effect to these variables is clarified in the notch sensitivity diagrams shown in Figs. 5 and 6.

In these diagrams the harmful effect of the notch is specified in terms of the fatigue strength reduction factor, K_f, which is defined as the ratio of the fatigue strength of the unnotched specimen to the fatigue strength (based on nominal stress calculations) of the notched specimen for the same life and same stress ratio.[3]

The first of the notch sensitivity diagrams, shown in Fig. 5, covers both the mild-notch and sharp-notch specimens. These were derived from "profile" curves (not shown) of K_f *versus* S_a or S_m, each of the profile curves covering a different alternating-mean ratio. The profile curves for each stress ratio were then projected onto the corresponding alternating-mean ratio line within the S_a *versus* S_m coordinate system of Fig. 5 to establish the contour curves representing the K_f surface.

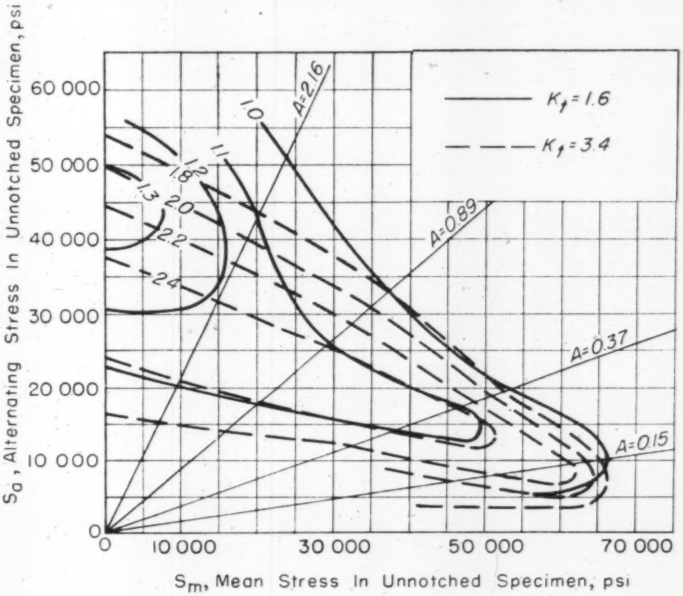

FIG. 5.—Fatigue Strength-Reduction "Contour" Curves for Notched Specimens of Aluminum Alloy 14S-T6 Showing K_f as a Function of S_a and S_m of the Unnotched Specimen.

The following observations may be made from Fig. 5. If the fatigue reduction factor K_f were reasonably independent of mean stress and dependent only on alternating stress, then the contour lines would be horizontal; whereas if K_f were dependent on mean stress and not on alternating stress, these contour lines would be vertical. In general, these contour lines are inclined, indicating the dependence of K_f on both alternating and mean stress. However, in some regions the lines are essentially horizontal whereas in other regions they are essentially vertical, indicating that either alternating or mean stress, respectively, may be the primary variable, depending on the conditions of test.

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[3] All data were procured and analyzed on the basis of a fixed stress ratio (rather than fixed alternating stress and variable mean stress or fixed mean stress under variable alternating stress). Thus, the ratio of $S_{max\ unnotched}$ to $S_{u\ notched}$ is the same as the corresponding S_a and S_m ratios, and it is not necessary in an analysis based on fixed stress ratios to specify which stress (S_{max}, S_a, or S_m) was used to compute K_f.

For the sharp-notch specimen, the highest K_f factor of 2.4 extends over a central region which includes alternating-mean ratios as low as 0.25. The maximum K_f observed (2.4) is considerably below the theoretical stress concentration factor of 3.4 for this specimen type.

The behavior trend of the curve for $K_t = 1.6$ specimens is about the same as that discussed above except that the maximum value occurs at a higher stress and the peak is concentrated more at the ∞ alternating-mean ratio line.

The curves for $K_t = 2.4$ (not shown) also displayed the same general pattern and were so shaped and positioned that a gradual transition could be observed in the characteristics of the mild notch through the intermediate to the sharp notch. Maximum K_f values observed were 2.5, 1.9, and 1.35 for the specimens whose K_t values are 3.4, 2.4, and 1.6, respectively.

Similar curves for 24S-T4 revealed characteristics very similar to those of 14S-T6 described above, except that maximum values were slightly higher. For 75S-T6, the same behavior characteristics were noted except that maximum values of K_f occurred at higher stress for the sharp notch and at lower stress in the case of the mild notch, and the maximum values were somewhat lower than for 14S-T6 (maximum observed K_f was 2.0 for the $K_t = 3.4$ specimens).

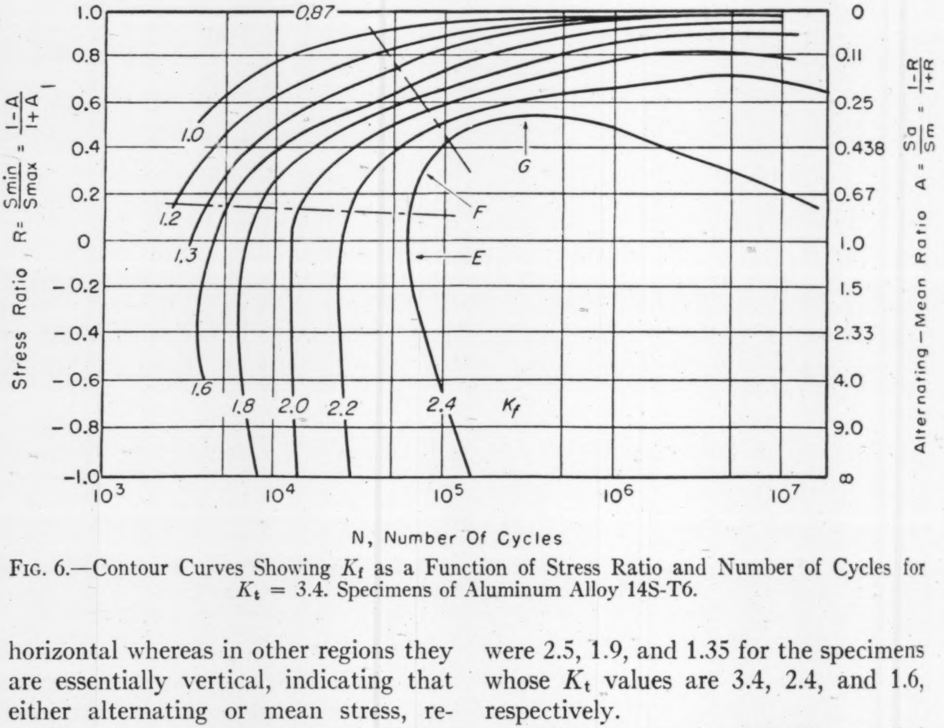

FIG. 6.—Contour Curves Showing K_f as a Function of Stress Ratio and Number of Cycles for $K_t = 3.4$. Specimens of Aluminum Alloy 14S-T6.

In order to clarify the rôle of length of fatigue life, a second type of notch sensitivity diagram is shown in Fig. 6. The contour lines shown in Fig. 6 for the K_f surface are constructed within stress ratio *versus* life N coordinates by projecting sets of profile curves of K_f *versus* life for each stress ratio. The maximum to minimum stress ratio R is used in Fig. 6 instead of the alternating-mean ratio A, because the range in R values from -1.0 to 1.0 (reversed stress to static stress) is more easily diagramed than the corresponding range of A values from zero to infinity. However, both scales are shown in this diagram for completeness.

Referring to Fig. 6, it is possible to divide the K_f surface into characteristic regions E, F, and G. In region E, in which the fatigue life is short and alternating-mean ratio A is large, the K_f contour lines are essentially vertical. This indicates that K_f is essentially independent of stress ratio and primarily dependent on the fatigue life. In region G, however, where the fatigue life is comparatively long and the alternating-mean ratio A is small, the contour lines are essentially horizontal, indicating that stress ratio rather than life is more critical. Region F, the transition region between E and G, is one in which both stress ratio and fatigue life are important.

The large slope in the K_f surface in region G, as indicated by the density of the K_f contour lines, again indicates that adding relatively little alternating stress in a notched member may greatly decrease the allowable preload.

There is a gradual transition in going from the severely notched specimen (shown in Fig. 6) to the mildly notched specimens (not shown), the three regions discussed previously being least apparent in the case of the mild notch. Also, the K_f peak occurs at higher alternating-mean ratio A and at somewhat shorter life in the case of the mild notch. It is therefore apparent that K_f varies significantly with both stress ratio and life and that the pattern of variation is dependent on the severity of the notch.

Similar diagrams were prepared for 24S-T4 and 75S-T6 but are not included. The diagrams for 24S-T4 were very similar to those of 14S-T6, and the corresponding curves for 75S-T6 were similar in general pattern though they varied somewhat in location of maxima, and the maximum values were lower than for 14S-T6 and 24S-T4.

Several attempts have been made in the past (**13, 14**) to analyze the over-all fatigue strength reduction factor K_f in terms of two more basic quantities: (*a*) the theoretical stress concentration factor K_t determined from the geometry of the notch, and (*b*) a material factor. A quantity sometimes used to represent the material factor is the dimensionless quantity q given by the expression $q = \frac{K_f - 1}{K_t - 1}$. On the basis of this definition q would normally lie between 0 (for a material that is extremely notch insensitive) to 1 (for a material which is extremely notch sensitive).

It is unlikely that a single material constant of the type q would adequately define the behavior of material at all stress ratios since both the static and fatigue properties may be involved to a varying degree. For similar reasons q is likely to be dependent upon fatigue life. Past work (**14**) indicates that q does vary considerably with factors other than material. Nevertheless, in the absence of a better measure of the material factor, the values of q were diagramed (**18**) within alternating and mean stress coordinates for the three type specimens $K_t = 1.6$, 2.4, and 3.4. Although the general shapes of the q surfaces for these types of specimen were similar in some respects, the specific values for q were generally quite different even under given stress conditions. In general, the sharp notch displayed the highest q factor over most of the stress-range field.

It is apparent that further work is required before a basic analysis can be made of the fatigue strength reduction factor.

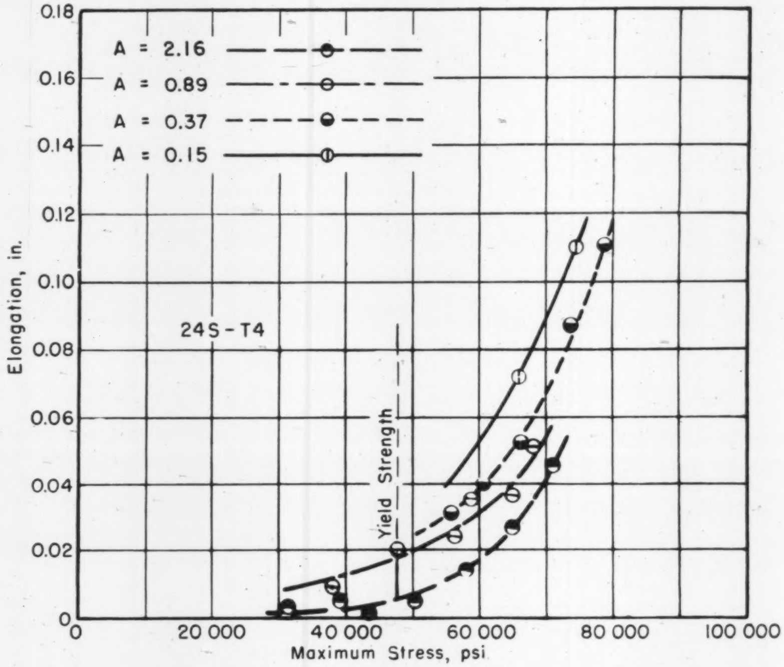

FIG. 7.—Elongation in Fatigue Specimens at Various Stresses and Alternating-Mean Ratios.

YIELD, CREEP, AND ELONGATION DURING FATIGUE TEST

It was observed that, for stresses in excess of the yield strength, there was considerable creep in the specimen during fatigue tests in which there was some applied mean stress. This yield, measured with a micrometer, occurred almost entirely during the early stages of the test. The fatigue machines used in this program had provision for automatically maintaining the desired mean stress even though the specimen elongated.

Total elongation was also determined by measuring the total length of the specimen before and after the test, the broken specimens, of course, being carefully matched at the fracture before measurement. Total elongation measured by this method compared favorably with the elongation measured by the micrometer method, although some difference due to elongation during final separation was sometimes apparent.

The total elongation of the specimens as a function of maximum stress and alternating-mean ratio is shown in Fig. 7. In general, elongation does not become significant until the maximum stress exceeds the yield strength. Above this limit elongation decreases with increasing stress ratio, becoming negligible under reversed stress conditions.

COMPARISON WITH DATA FROM OTHER SOURCES

A comparison was made of the fatigue data obtained in this program with those from other published sources (1–9, 16). In general the agreement was good for 14S-T6 and 24S-T4, as regards both fatigue strength and notch sensitivity. However, the comparison of 75S-T6 data reveals rather serious divergence. The Minnesota data is as much as 50 per cent below the comparison data at 10^5 cycles and shows an even greater discrepancy at 10^7 cycles. Tests conducted at the Aluminum Research Laboratories (16) on specimens from the same batch of material as tested in this program yield fatigue strengths significantly below average values. The ARL data fall between Minnesota data and those from other sources. Comparative curves indicated that, though the static strengths are reasonably uniform, there is a wider variation in fatigue strengths of 75S-T6 than for the other aluminum alloys.

SUMMARY AND CONCLUSIONS

A series of axial stress fatigue tests were undertaken on 14S-T6, 24S-T4, and 75S-T6 to determine the effect of various combinations of alternating and mean stress on fatigue life. The stress range from reversed stress (from tension to equal compression) to static tension was covered in several alternating-mean ratios. Four types of specimens were studied, an unnotched type and three circumferentially notched types having theoretical stress concentration factors of 1.6, 2.4, and 3.4. Stress combinations were varied to produce fatigue failure in the range from 10^4 to 10^8 cycles.

The basic fatigue data are presented in the form of *S-N* curves and stress range diagrams for 14S-T6, and results for the other materials are described. Since the ratio of unnotched to notched fatigue strengths varied considerably with alternating-mean ratio and stress magnitude (or life), the fatigue strength reduction factor was plotted as a K_f surface within two coordinate systems: (*a*) alternating stress *versus* mean stress, and (*b*) stress ratio *versus* number of cycles to failure. In addition, the data was partially analyzed to determine the validity of the notch sensitivity index q as an indication of the material factor.

The fatigue data procured in this program were compared with published data on similar materials.

The following conclusions are based on the fatigue and notch sensitivity data presented in this paper.

1. Static tension tests performed on two types of unnotched specimens and three types of notched specimens indicate an approximate 25 per cent increase in strength up to a theoretical notch severity of 2.5, beyond which a significant reduction occurs. This trend may be partially explainable on the basis of two opposing factors introduced by the notch: the weakening effect of stress concentrations *versus* the strengthening effect which the notch offers in confining the reduction in area preceding fracture.
2. Based on limited tests not reported in this paper, the 100-hr stress-rupture strength of all specimen types at room temperature are approximately the same as the static tensile strength.
3. The axial stress fatigue properties of unnotched and notched 14S-T6 are practically equal to those of 24S-T4 at all stress ratios from static tension to reversed stress. The properties determined in this program checked prior work within practical limits.
4. Even though the static properties of the 75S-T6 used in this program are normal, the fatigue properties are abnormally low and significantly below those of 14S-T6 and 24S-T4. In general, there appears to be greater divergence

in fatigue properties of different lots of 75S-T6 than in 14S-T4 and 24S-T4.

5. The stress range diagrams (S_a *versus* S_m for fixed number of cycles to failure) display extreme flatness and upward concavity for notched specimens. In the sharply notched specimens, for example, adding only 15 per cent alternating stress reduces the allowable mean load by an average of approximately 70 per cent. Thus, small magnitude vibrations frequently encountered in service, and sometimes ignored as being insignificant, may affect greatly the mean load carrying capacity of notched parts exposed to a large number of fatigue cycles.

6. Stress range data for 14S-T6 plotted on a completely unitless basis (ordinate and abscissa intercept arbitrarily made equal to one for all lives) fall within bands for each specimen type. For the unnotched and mildly notched specimen, a straight line joining the two unity values provides a reasonable approximation, which in most cases is somewhat conservative. In the sharply notched specimen, however, the band is S-shaped such that the data fall significantly below the straight line relationship for high alternating-mean ratios and somewhat above for low alternating-mean ratios. The same general patterns are evident in the 24S-T4 data except the band width is somewhat larger. For 75S-T6 the band width for the data on unnotched specimens is still wider and the long-life points fall considerably below the straight line relationship. For all materials in a severely notched condition, the data fall significantly below the straight line relationship at high alternating-mean ratios.

7. The fatigue strength reduction factor K_f plotted as a surface within an alternating stress *versus* mean stress coordinate system reveals, in general, contour lines which are inclined. This means that, in general, both alternating and mean stress significantly affect K_f, although in some regions one type of stress may be more critical than the other.

8. For the severely notched specimens at high alternating-mean ratios (A above 0.25), contour lines are reasonably straight and have an inclination with the horizontal between approximately 10 to 30 deg. At low ratios in the fatigue failure region, the slope of the K_f surface is extremely sharp (contour lines close together), which indicates that adding relatively little alternating stress to a high mean load greatly increases notch sensitivity. The peak of the K_f surface extends over a narrow plateau, covering alternating-mean ratios from infinity to approximately 0.25. The maximum K_f values observed are 2.4, 2.6, and 2.0 for 14S-T6, 24S-T4, and 75S-T6, respectively, compared with a theoretical notch sensitivity factor for the severely notched specimens of 3.4.

9. The specimens of 14S-T6 and 24S-T4 with the intermediate notch ($K_t = 2.4$) display approximately the same characteristics as those of the sharp notch, except that the effects are not so pronounced. The maximum K_f occurs along a plateau in the K_f surface extending from alternating-mean ratio ∞ toward a ratio of 0.15 at low stress magnitude. Maximum values of K_f are 1.8 for 14S-T6 and 2.2 for 24S-T4.

10. The mildly notched specimens display approximately the same type of K_f surface except that the peak is confined to alternating-mean ratios near infinity. The maximum K_f values are 1.3 for both 14S-T6 and 24S-T4 and 1.2 for 75S-T6 compared to a theoretical value of 1.6.

11. In order to more clearly reveal the effects of specimen life, the K_f surfaces were also plotted for each material within a stress ratio *versus* number of cycles to failure coordinate system. Here again the slopes of the K_f contour lines indicate

that both stress ratio and life may be of critical importance, depending on the region. For the sharp-notch specimens, a circularly shaped plateau appears in the K_f surface in the region of high alternating-mean ratio A and long life. For short fatigue lives the contour lines are essentially horizontal, indicating that stress ratio is considerably more critical than life. Thus, the addition of small alternating stress to a high mean load critically affects the fatigue strength reduction factor only at long fatigue life. The intermediate-notch and mild-notch specimens displayed somewhat similar behavior.

12. Plots were made of the notch sensitivity index q for the three types of notch specimens within a mean stress *versus* alternating stress coordinate system to determine if q is independent of notch geometry. As has been established in the past, q is greatly dependent on notch geometry and is not a true material factor even at a fixed alternating-mean ratio.

13. The elongation of fractured specimens at various alternating-mean ratios plotted against maximum stress indicates that the elongation is generally small at all ratios if the crest stress is below the static yield strength. However, when the crest stress exceeds the yield strength, the elongation becomes increasingly large with increasing crest stress and decreasing alternating-mean ratio.

Acknowledgment:

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The following University of Minnesota personnel assisted in the examination of materials and testing of the specimens: F. W. DeMoney, and H. Binder performed the metallographic work, and Paul M. Kemmer and S. H. Myhre conducted most of the tests.

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DISCUSSION

Mr. Joseph Marin.[1]—I should like to ask Mr. Lazan if anything has been done on the prediction of the mean-variable stress fatigue strength relation for notched bars based on the unnotched specimen behavior. Such a prediction would include a consideration of the influence of combined stresses.

Mr. B. J. Lazan (*author*).—We have made no attempt beyond using Neuber's relationship to correlate the notch and unnotched fatigue strength. One factor involved in such a correlation is whether to apply the stress concentration factor on the alternating stress only, or on both the alternating and mean stress. As indicated in the figures which show K_f as a function of mean and alternating stress, this is not a simple problem.

Mr. Harold Hessing.[2]—In Fig. 1 no point is plotted for the 75S-T6 curve at the stress concentration factor 2.4. Does this actually represent lack of data?

Mr. Lazan.—No point was procured for 75S-T6 at $K_t = 2.4$. However, the trend established by the other two materials is well defined; therefore, the 75S-T6 curve is probably not far off.

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[2] Metallurgist, Department of the Navy, Washington, D. C.

AN INVESTIGATION OF THE EFFECTS OF OVERSTRESS ON THE FATIGUE CHARACTERISTICS OF CERTAIN WROUGHT SHEET MAGNESIUM ALLOYS*[1]

BY F. S. WILLIAMS[2] AND J. VIGLIONE[2]

SYNOPSIS

A number of studies were conducted at the Naval Air Experimental Station in an attempt to determine to what extent the mechanical properties of magnesium alloys used in stressed structures in naval aircraft were affected by service stresses. These studies revealed the need for a comprehensive investigation evaluating the effects of the controlled prestress upon the fatigue strength of wrought magnesium alloys.

In this investigation the effects of overstressing of cold-rolled AZ61A and AZ31A magnesium alloys have been determined and correlated with the location of the damage line. It was found that these magnesium alloys were damaged in fatigue by understressing as well as by overstressing, whenever the prestress condition was above the damage line. The damage effect was greatest at the higher prestress levels, leading to the conclusion that the amount of damage is more a consequence of the magnitude of the prestress than of the number of stress applications. It was also shown that the fatigue strength and the fatigue life of these alloys was neither raised nor extended by prior understressing below the damage line. The tensile properties seemed to be unchanged by prestressing occurring either above or below the damage line.

Fatigue is one of the most important contributing factors causing sudden mechanical failure of highly stressed structural aircraft component members while in service. It is reasonable to assume that the structural members are being damaged as a result of service stressing occurring during flight. However, the extent of the damage cannot be detected or ascertained until the visible cracks are formed, and when this happens the remaining life before actual failure is relatively brief. There is no known feasible non-destructive inspection method of determining damage occurring during the precrack stage. Although a considerable amount of experimental research has been done to obtain data applying to the fatigue life of a specific part or structure used in a particular machine, there is little known fundamental information available that can ultimately lead to an explanation of the behavior of basic materials under repeated stress, particularly as concerns quantitative damage prior to the appearance of a visible crack.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] The opinions or assertions expressed are the private ones of the writers and are not to be construed as official or reflecting the views of the Navy Department or the naval service at large.

[2] Head and Metallurgist, respectively, Metallurgical Division, Aeronautical Materials Laboratory, Naval Air Experimental Station, Philadelphia, Pa.

TABLE I.—FATIGUE STRENGTH OF SPECIMENS OF SAMPLES FROM DIFFERENT LOCATIONS OF WING PANELS.

Location	Fatigue Strength Based on 50 Million Cycles			
	AZ61A Alloy		AZ31A Alloy	
	psi	Reduction, per cent	psi	Reduction, per cent
Group 1	10 800	27.1	12 250	23.0
Group 2	11 250	23.8	12 250	23.0
Group 3	13 250	10.2	11 500	28.0
Group 4	13 750	6.8	14 000	12.0
Group 5	13 500	8.5	13 500	16.0
Group 6	13 500	8.5	13 000	19.0
Unstressed metal	14 750	...	16 000	...

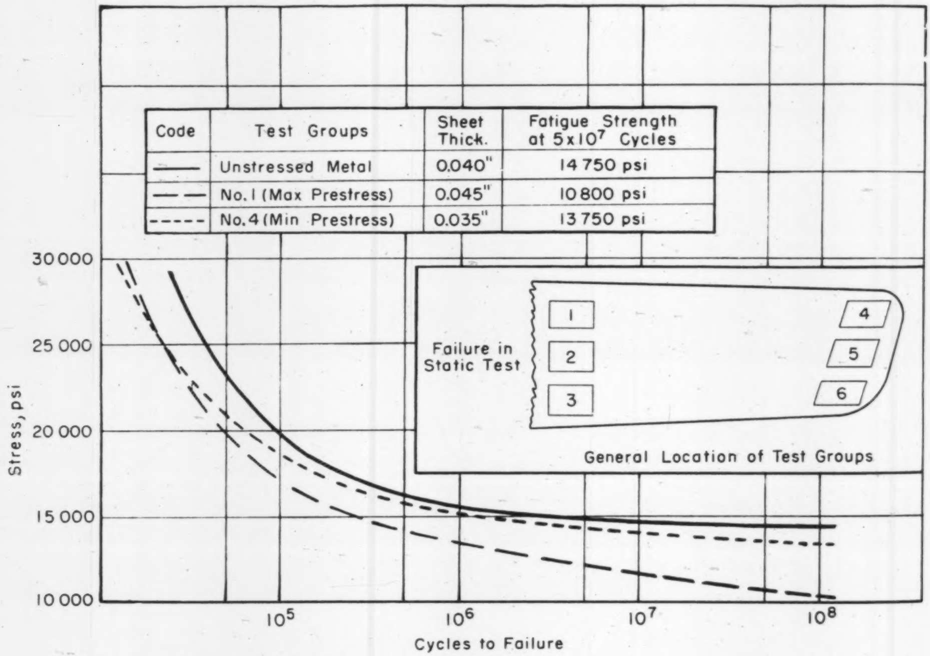

FIG. 1.—Effect of Flight Stresses on Fatigue Strength AZ61A Magnesium Alloy.

The Bureau of Aeronautics, aware of the necessity for compiling pertinent fundamental information on the mechanical properties of light non-ferrous alloys used in stressed structures which have been in service for a considerable length of time, authorized the Naval Air Experimental Station to conduct several metallurgical examinations of some wing panels. It was found that there was little or no change, from the original, in tensile, compressive, and hardness properties of the AZ61A and AZ31A magnesium alloy skin and extruded formers removed from the wing panels. However, the fatigue strength was reduced as compared to the fatigue strength of unstressed virgin magnesium alloy, the greatest reduction occurring in samples removed from inboard sections adjacent to the area of maximum prestress. Independent tests conducted by the Dow Chemical Co. (**1**)[3] on similar samples removed from the same wing panels generally confirmed the results obtained at the Naval Air Experimental Station that fatigue strength has been impaired by service use while static strength had not. Summarized results of the tests of the wing panel specimens are shown in tabular form in Table I, with a specific typical example illustrated in Fig. 1.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 882.

It could not be stated for certain what effects, quantitatively, specific service stresses had had upon the magnesium sheet material removed from the wing panels, since neither the type, magnitude, nor the number of service stress applications were exactly known. It was believed that a better approach to the problem would be constituted by a comprehensive study evaluating the effects of controlled prestress on the mechanical properties of wrought magnesium sheet material whose mechanical properties could be accurately determined in the unstressed condition. Thus, a test program was initiated to study the effects of overstress on the fatigue strength of AZ61A and AZ31A magnesium alloys, with special attention to overstressing at levels near the maximum condition of stress and low number of reversals of stress. Such a study would attempt to correlate laboratory fatigue test results with service history in hopes of finding a reasonably satisfactory means of predicting probable service life, or would attempt at least to determine a trend which might ultimately develop as a basis for additional design criteria for light alloys.

Although AZ61A sheet material is no longer used in new naval aircraft construction and is not commercially obtainable, a sufficient quantity was immediately available at the Naval Air Experimental Station for tests and for use in orienting the scope and details of the later and more important phases of the project when studying the effects of prestress in AZ31A.

Of course, it is realized that the effect of prestress on the fatigue life of magnesium alloys as performed in the laboratory is not exactly analogous to the effect of service stress in structural members of aircraft component parts. In the laboratory, the maximum intensity of stress per cycle is practically constant during the life of the specimen, whereas the aircraft structural parts are subject to repeated applications of loads of varying magnitudes and random fluctuations, where both the maximum and minimum intensity of load during successive cycles are subject to variations. An airplane wing, for example, must support not only the weight of the plane, which is a steady load, but also a relatively light fluctuating load due to steady gusts when flying at cruising speeds and a high fluctuating load due to either large and sudden wind gusts or dives, which occur somewhat infrequently. Thus, in the case of some aircraft structural component parts, we have the possibility of a large number of applications of a relatively small load and a small number of applications of a large load, all superimposed randomly upon a steady load. Although the laboratory tests, in this investigation, did not simulate actual fluctuating service stresses, the primary objective of the investigation was to

TABLE II.—CHEMICAL COMPOSITION AND MECHANICAL PROPERTIES.

	AZ61A	AZ31A
CHEMICAL COMPOSITION, PER CENT		
Aluminum	5.8 to 7.2	2.5 to 3.5
Manganese	0.15	0.20
Zinc	0.4 to 1.5	0.7 to 1.3
Silicon (min)	0.3	0.3
Copper (max)	0.05	0.05
Nickel (max)	0.005	0.005
Iron (max)	0.005	0.005
Others	0.3	0.3
Magnesium	Remainder	Remainder
MECHANICAL PROPERTIES		
Tensile strength, psi	47 300	41 100
Yield strength,[a] psi	36 000	31 300
Elongation in 2 in., per cent	14.5	14.5
Fatigue strength, psi	13 000	14 000

[a] Yield strength based on 0.2 per cent offset method.

accumulate fundamental damage data which could be used as a basis for a more complicated study under a laboratory environment that more nearly approached actual service conditions.

MATERIALS AND PROCEDURES

The AZ61A and the AZ31A magnesium alloys were supplied in sheet form, 0.064-in. nomnial thickness. The AZ61A alloy had a chrome pickled finish and the AZ31A alloy was furnished in the oiled condition, with nominal chemical composition and mechanical properties, respectively, as shown in Table II.

Mechanical property values for the AZ61A alloy were determined at the Naval Air Experimental Station; all tensile property values are the average of at least two specimens. Similar values for the AZ31A alloy were reported by the Dow Chemical Co. (Certification of Properties). In both cases the fatigue strength characteristics were determined at the Naval Air Experimental Station and are based on 100 million cycles.

Sheet flexure fatigue tests were carried out on constant amplitude fatigue testing machines, capable of reproducing all ranges of bending stresses by merely shifting the gripping vise from the neutral position and by appropriately varying the degree of bending by means of the eccentric variable throw crank. With the gripping vise in the neutral position the specimen is subjected to completely reversed bending stresses, the range of stress used throughout this investigation. A preliminary study indicated that the test was substantially one of uniform stress, until cracks developed, and that no adjustment in eccentric setting was necessary to maintain constant stress on the specimen during the progress of the test.

The terms overstressing and understressing, as normally used, are related to stresses above or below the fatigue limit, which is defined as the highest unit stress whose repeated application can be indefinitely endured without failure. These terms are somewhat arbitrary for most non-ferrous alloys, which have no definite fatigue limit. Materials which fall in this category, such as magnesium alloys, may be studied by subjecting the metal to repeated stress cycles short of failure and then subsequently raising or lowering the stress with respect to the prestress and observing an increase or a decrease in specimen life, as compared with that of the metal not prestressed.

Damage and its effect on the fatigue properties of the magnesium alloys was investigated by (1) determining a damage line, (2) determining quantitative damage, (3) comparing life of understressed and overstressed specimens with that of specimens not prestressed, and (4) verifying the damage line, as follows:

1. The damage line was obtained by overstressing specimens for a given number of cycles at some stress above the fatigue strength for 100 million cycles (as determined by test) and subsequently retesting the same specimen at this predetermined fatigue strength. Those retested specimens failing short of the 100 million cycles were considered damaged, whereas those which did not fail at 100 million cycles were presumed to be undamaged.

2. Quantitative damage determination was accomplished by subjecting an entire group of specimens to repeated stress all at the same stress level and number of cycles, and subsequently determining the fatigue strength of the prestressed material at 100 million cycles. If the fatigue strength of the prestressed group was lower than that for the virgin

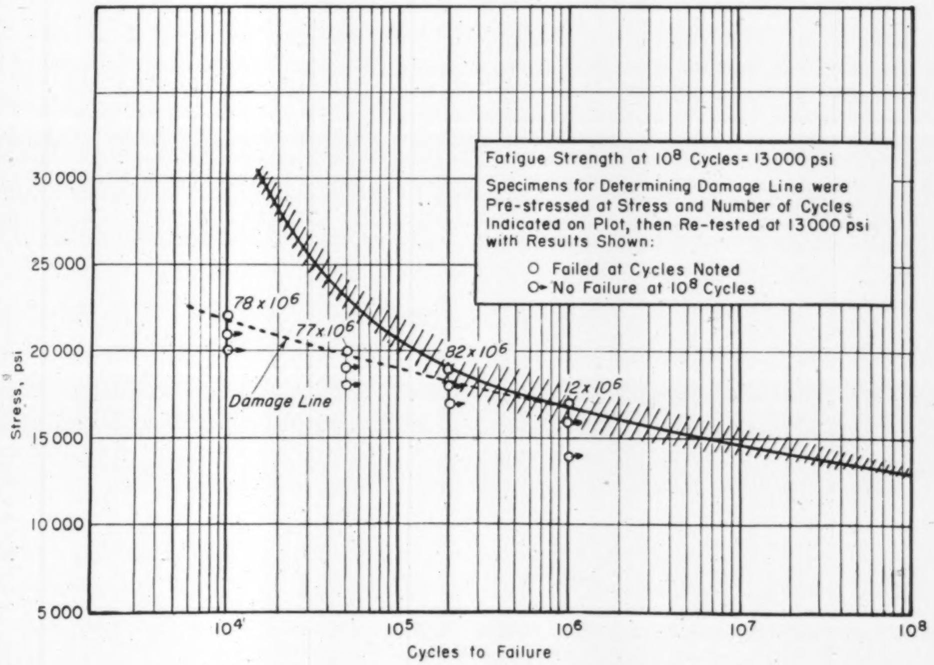


FIG. 2.—AZ61A Magnesium Alloy Scatter Band and Damage Line.

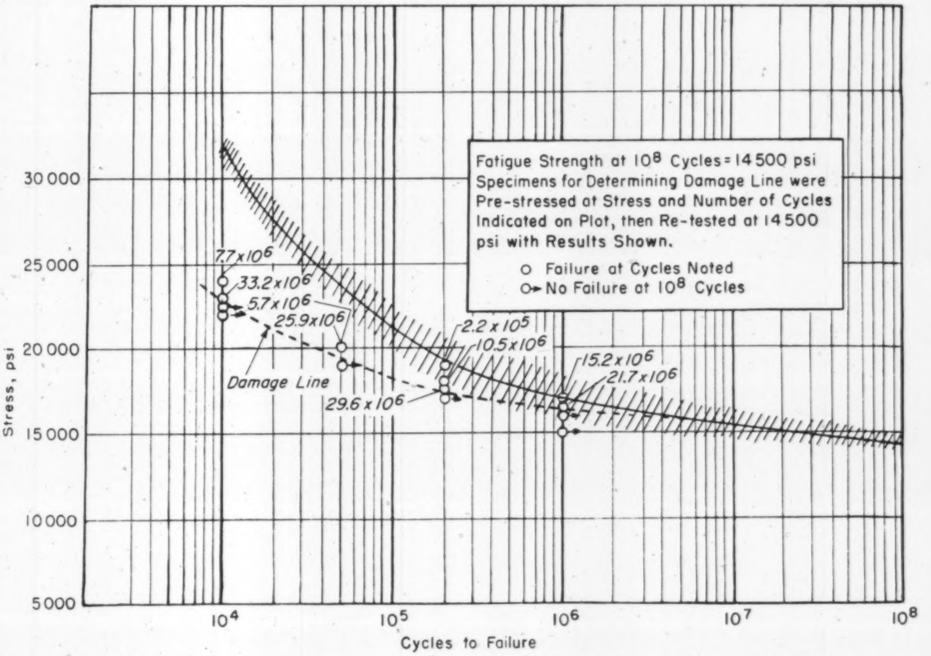


FIG. 3.—AZ31A Magnesium Alloy Scatter Band and Damage Line.

material, the group was presumably damaged. Similarly, if the fatigue strength of the prestressed group proved to be either higher than or equal to that of the virgin metal, then this group was considered to have been either improved or undamaged, respectively.

3. The effect of understressing and overstressing was evaluated by subjecting specimens to cycles of stress short of failure and subsequently retesting at higher and lower stresses and comparing their fatigue lives with those of specimens of virgin material tested at these higher and lower stresses.

4. To verify the damage line, three groups of four specimens each were prestressed (short of failure) for the same number of cycles but at three different stress levels, respectively, below the damage line. These prestressed specimens were tested at a single stress, which for failed virgin material resulted in points on the *S-N* diagram above the damage line. Evidence of no change in the life of the specimens at the latter test stress, as compared with the specimen life shown by the curve for the virgin material, was assumed to indicate satisfactory determination of the damage line.

In an effort to determine what effects, if any, prestressing had upon the tensile qualities of the alloys, a number of tenison fatigue specimens were overstressed and understressed in a tension fatigue testing machine, above and below the damage line, respectively. Standard tension specimens were then machined from these prestressed fatigue specimens, tested in static tension and the results compared with those of the unstressed control group. To insure damage, two specimens of each test group were tested to failure, thus establishing a correlation between tension and flexure fatigue tests and appropriately prestressing the remainder of the specimens short of failure. In this case, prestressing was done on a tension fatigue testing machine in order to obtain prestressed fatigue specimens sufficiently large to convert into standard tension specimens, a procedure not otherwise possible if the fatigue specimens had been prestressed in a flexure fatigue testing machine.

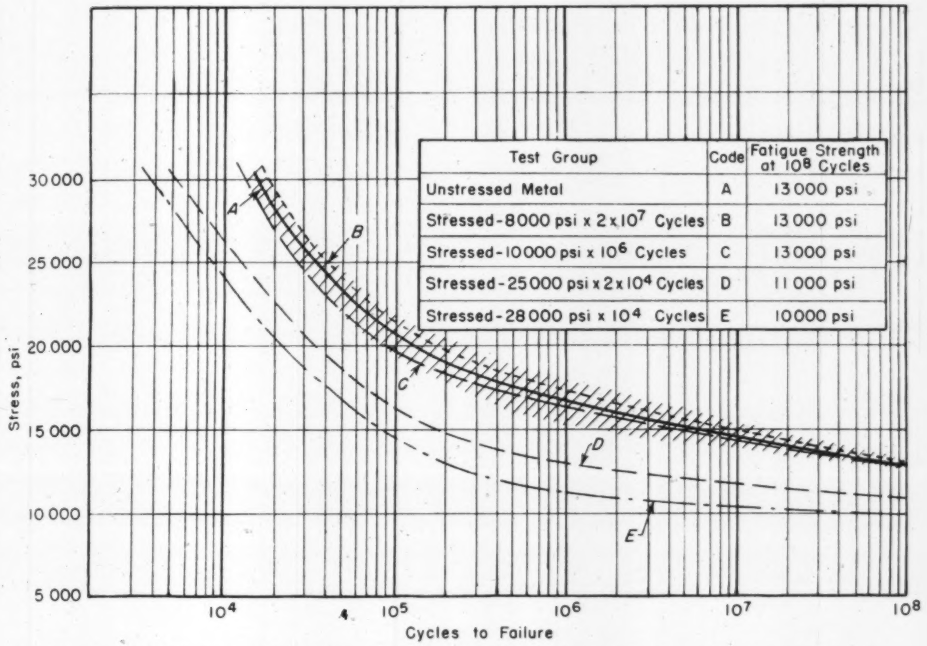
TABLE III.—EFFECT OF PRESTRESS ON FATIGUE STRENGTH.

Prestress		Fatigue Strength, psi	Percentage Reduction from Unstressed Metal
Stress, psi	Cycles		
AZ61A Alloy			
28 000	10 000	10 000	23.0
25 000	20 000	11 000	15.0
10 000	1 000 000	13 000	0
8 000	20 000 000	13 000	0
Unstressed metal		13 000	0
AZ31A Alloy			
28 000	10 000	12 000	17.3
25 000	20 000	12 500	13.8
20 000	50 000	12 500	13.8
15 000	1 000 000	14 500	0
10 000	10 000 000	14 500	0
8 000	30 000 000	14 500	0
5 000	100 000 000	14 500	0
Unstressed metal		14 500	0

NOTE.—Each test group consists of eight specimens.

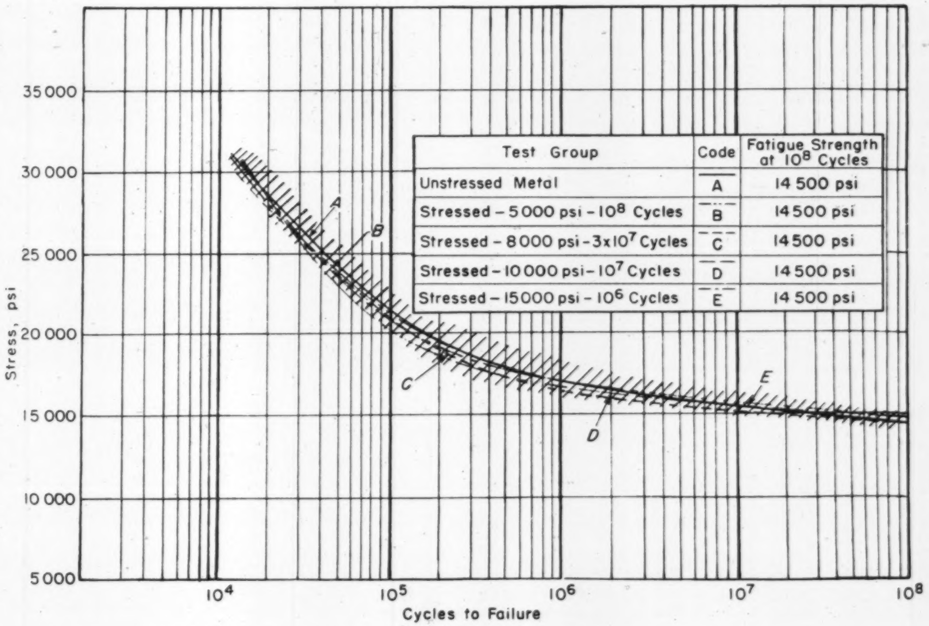
RESULTS

The *S-N* scatter band for the AZ61A and the AZ31A magnesium alloys, together with the damage line, are shown in Figs. 2 and 3, respectively. The test plot points on this and other *S-N* diagrams have been omitted for reasons of clarity to permit presentation of other data which can best be evaluated by visual comparison with the scatter band. It may be observed that the damage lines pass through the lower portion of the scatter band. The results of tests to determine quantitatively the damage



Test Group	Code	Fatigue Strength at 10^8 Cycles
Unstressed Metal	A	13 000 psi
Stressed-8000 psi x 2×10^7 Cycles	B	13 000 psi
Stressed-10000 psi x 10^6 Cycles	C	13 000 psi
Stressed-25000 psi x 2×10^4 Cycles	D	11 000 psi
Stressed-28000 psi x 10^4 Cycles	E	10 000 psi

FIG. 4.—AZ61A Magnesium Alloy Effect of Controlled Prestresses on Fatigue Strength.



Test Group	Code	Fatigue Strength at 10^8 Cycles
Unstressed Metal	A	14 500 psi
Stressed – 5 000 psi – 10^8 Cycles	B	14 500 psi
Stressed – 8 000 psi – 3×10^7 Cycles	C	14 500 psi
Stressed – 10 000 psi – 10^7 Cycles	D	14 500 psi
Stressed – 15000 psi – 10^6 Cycles	E	14 500 psi

FIG. 5.—AZ31A Magnesium Alloy Effect on Controlled Prestresses on Fatigue Strength.

arising from prestressing at different values of prestress and cycles of stress applications are summarized in Table III, expressed as fatigue strengths at 100 million cycles, and are shown in Fig. 4 for the AZ61A alloy and Figs. 5 and 6 for the AZ31A alloy.

The results of re-evaluating specimens prestressed within the damage zone, Figs. 7 and 8, for the AZ61A and the AZ31A alloys, respectively, indicate that the specimens were slightly damaged and failed to perform as well as the virgin material when evaluated at stress levels above or below the prestress value; that is, the material was damaged by this relative understressing as well as by the overstressing. This behavior appears to be adequately explained by the fact that the prestress exceeded the minimum stress to cause damage, as predicted from the damage line. Groups of specimens prestressed one million cycles at different stress levels (10,000, 13,000, and 15,000 psi) below the damage line and afterwards evaluated at 18,000 psi had fatigue lives that ranged, without any systematic pattern, across the scatter band for the virgin material at that stress. These data are presented in Figs. 7 and 8. While no definite strengthening effect could be attributed to the understressing, the absence of any pronounced noticeable damage resulting from the prestress serves to verify the location of the damage line.

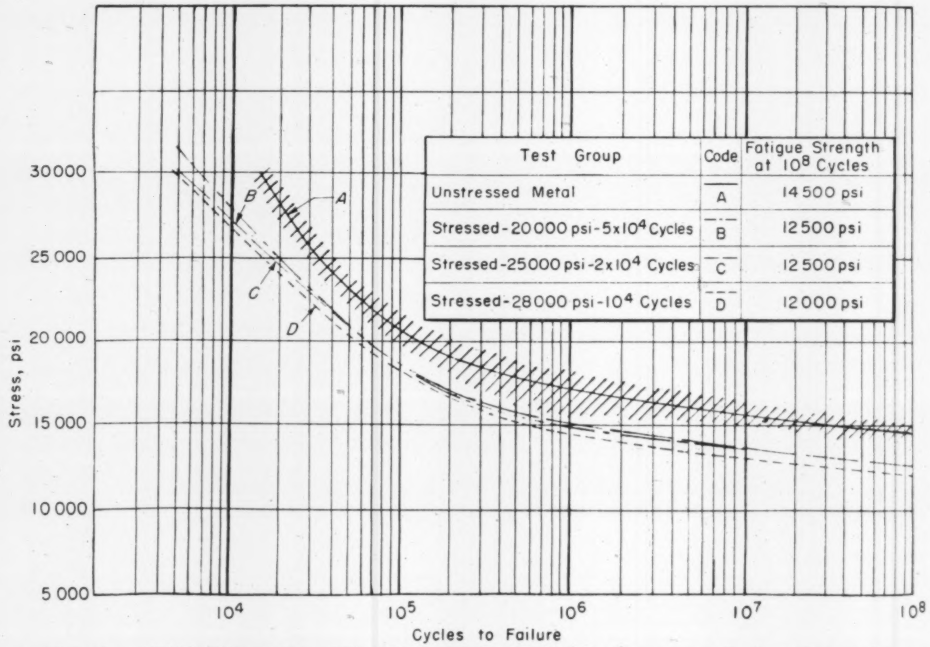


FIG. 6.—AZ31A Magnesium Alloy Effect of Controlled Prestresses on Fatigue Strength.

The results of the effects of fatigue prestress on the static tensile characteristics of the magnesium alloys are listed in Table IV. It is apparent that the tensile properties of the prestressed specimens (whether prestressed above or below the damage line) were unchanged from those of the unstressed virgin specimens.

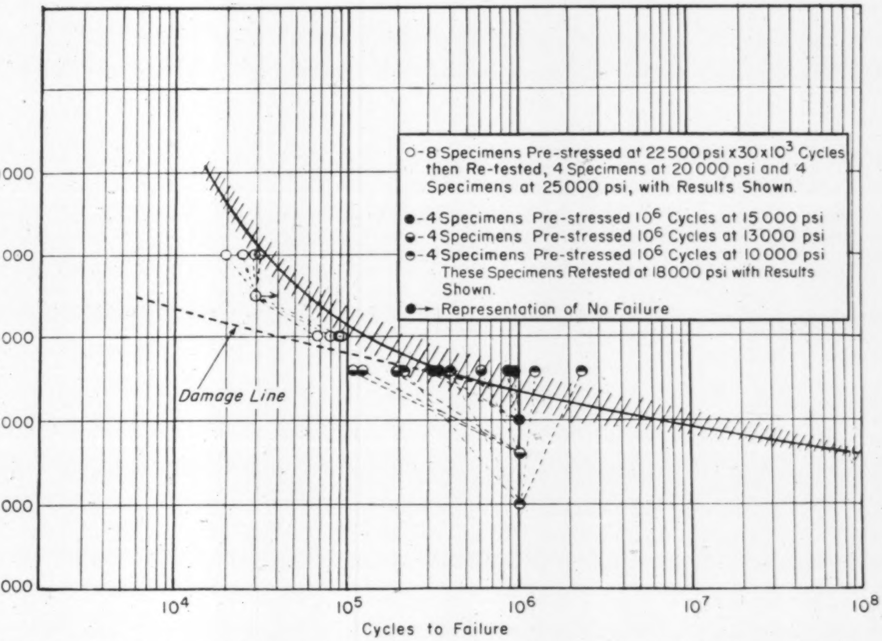


FIG. 7.—AZ61A Magnesium Alloy Damage Line *versus* Overstressing and Understressing.

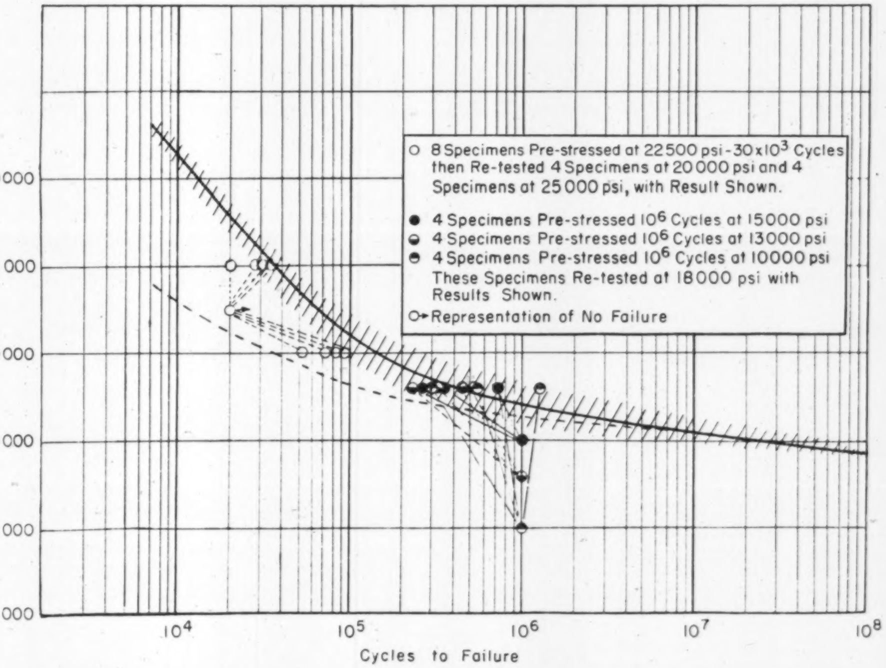


FIG. 8.—AZ31A Magnesium Alloy Damage Line *versus* Overstressing and Understressing.

DISCUSSION OF RESULTS

A literature survey failed to reveal any information on the subject of damage lines for magnesium alloys. Johnson and Oberg (2) reported that aluminum alloy propellers retired from service (after the number of hours that damage line data had indicated as proper time for retirement), and then tested on a test rig, had much shorter lives than new unused propellers. After considerable study, Johnson also made the very important observation that the damage line for specimens from a forged aluminum propeller closely parallels the lower boundary of the fatigue scatter band. However, it is to be noted that the damage lines for the magnesium alloys investigated depart rather sharply from the scatter band for fatigue lives less than 100,000 cycles and 1,000,000 cycles for the AZ61A and AZ31A magnesium alloys, respectively.

TABLE IV.—TENSILE CHARACTERISTICS AFTER PRESTRESS.

Prestressed		Tensile Strength,[a] psi	Yield Strength,[b] psi	Elongation in 2 in., per cent
Stress, psi	Cycles			
		AZ61A ALLOY		
30 000	10 000	46 500	37 300	10.5
25 000	20 000	46 900	35 700	13.5
23 000	50 000	46 300	35 600	11.0
19 000	200 000	46 300	36 600	9.0
16 000	1 000 000	48 300	38 200	12.0
Unstressed metal		47 300	36 000	14.5
		AZ31A ALLOY		
30 000	10 000	41 700	31 700	13.0
25 000	30 000	41 800	30 800	14.5
23 000	50 000	41 900	29 800	15.5
20 000	100 000	41 000	28 600	16.5
16 000	1 000 000	42 100	30 400	15.0
Unstressed metal		41 800	29 900	16.5
Unstressed metal[c]		41 400	31 300	14.5

[a] All tensile property values are average of at least two specimens, except for Dow Chemical Co. figures.

[b] Yield Strength based on 0.2 per cent offset method.

[c] Reported by Dow Chemical Co. (Certification of Properties).

Specimens which had been prestressed at low stress levels and great number of cycles did not have reduced fatigue life at 100 million cycles, nor a marked reduction in fatigue life at the high stress levels; rather these low prestressed groups of specimens had *S-N* curves that closely followed the lower edges of the scatter band for the unstressed material. In contrast to this, those specimens prestressed at high stress levels and few cycles either had lower fatigue strengths at 100 million cycles or had shortened lives when evaluated at either higher or lower stresses. This behavior can be explained by consideration of the prestress condition with respect to the damage line. These prestress conditions (combination of cycles and stress) falling above the damage line, deleteriously affected the subsequent fatigue performance of the test specimens. Conversely, no damage was observed as a result of prestress conditions falling below the damage line. These results serve to verify the location of the damage line.

It is of interest to observe that there appears to be no instance in which the magnesium alloys are strengthened by understressing, in marked contrast to the behavior of the carbon or low-alloy constructional steels which are strengthened by understressing, as reported by Battelle (3). The data presented herein indicate that understressing or overstressing magnesium alloys is detrimental only if either occurs above the damage line, but results in neither decreasing nor increasing the specimen fatigue life if occurring below the damage line.

It is to be noted that the prestressing of the tension specimens was done in an axial load type fatigue testing machine with stress range of minimum tensile to maximum tensile for a stress ratio of 0.25, whereas the prestressing of the fatigue specimens was done in a flexure fatigue testing machine with a stress range of maximum compression to maximum tension, for a stress ratio of −1. As a consequence of this, the effect of prestressing the tension specimens was not as great as the effect of prestressing the flexure fatigue specimens, for the same stress levels. But the higher prestress levels of the tension fatigue specimens were chosen to fall considerably above the damage line, close to the *S-N* curve, after a correlation between tensile and flexure fatigue values was established, to insure that damage did take place.

The damaging effect brought about by certain prestress on the magnesium alloys investigated can best be conveniently explained by the cumulative damage (**4**) theory which theorizes that fatigue damage and eventual failure occur from using up of some percentage of the initial fatigue life of the material under varying amplitudes of stress. It may be said that during each cycle of stress, the material is progressively undergoing damage until failure occurs. With prestress, part of the life corresponding to the virgin *S-N* curve for that stress, is used up to initiate damage on a submicroscopic scale long before any signs of damage become visible on a microscopic scale. If fatigue damage is considered in this light, it is apparent that prestress, either service or controlled, would have a tendency to translate the original *S-N* curve leftward an amount depending upon the amount of damage, that is, the magnitude of the prestress and the number of stress applications. This would mean that some of the original fatigue life at high stress levels had been expended without an appreciable change in the fatigue life at low stress levels, particularly at the fatigue strength corresponding to 100 million cycles, which was arbitrarily used as a criterion for comparison.

In the studies reported in this paper, it seems that all of the *S-N* curves of the groups prestressed at low stress levels, that is, up to 10,000 psi for the AZ61A alloy and 15,000 psi for the AZ31A alloy, the *S-N* curves were translated slightly to the left, so that the comparative fatigue strength at 100 million cycles remained unchanged, although fatigue life at higher stress levels was somewhat reduced. However, the *S-N* curves of the higher prestressed groups took the form of a downward shift which denotes a reduction in fatigue life as well as a decrease in the fatigue strength of 100 million cycles. As postulated by one investigator (**1**), it is possible that the "percentage-life-used" (cumulative damage theory) is applicable over the low prestress ranges, but that higher prestress tends to produce "reduced-endurance-limit" type of damage. An additional explanation offered would be that the "reduced-endurance-limit" type of damage is the primary one and always present, but that continued application of low stress cycles "coaxed" some of the life back into the material to obscure some of the damage taking place at the lower stress levels.

An attempt was made to analyze statistically the test data, since it appears to be fairly well established that both fatigue life and fatigue limit values are statistical quantities. An analysis of the problem indicated that it was impossible to give a significance test to the statistical coefficients with so few data. It is considered that at least 20 combinations of applied cyclic stress and corresponding fatigue strength values are necessary in order to obtain a good estimate, by statistical analysis, of the effects of overstress and understress on these alloys. This requirement would be extremely difficult to meet within the scope of this investigation, which was to be more of a qualitative exploratory nature intended only to indicate trends and effects, to be followed by similar and more comprehensive investigations if the conclusions so warranted.

It is appreciated that the scatter of fatigue tests is inherently large and that the range of scatter can be reduced by extreme care in selection and preparation of specimens, but essentially fatigue data is highly statistical in character (5). It has been shown by other investigators that fatigue properties exhibit a rather large statistical variability, larger than commonly appreciated. Six experimental factors were examined by Ransom and Mehl (6) for their possible contribution to this observed variability. In no case was there conclusive proof that an experimental factor contributed to the scatter, leading to the conclusion that fatigue is truly a statistical phenomena.

It can be said that this latter consideration has not received enough attention in this investigation, but it is again emphasized that the nature and scope of this study precluded testing large quantities of specimens for proper statistical analysis. If further investigation along these lines is contemplated, it would be desirable to concentrate all efforts on one alloy to obtain a complete and adequate set of data and plan in advance for testing at predetermined values of cyclic prestress applications designed to give best statistical analysis.

CONCLUSIONS

The wrought magnesium alloys investigated were shown to have very definite damage lines which passed through the lower part of the scatter band at 100,000 cycles in the case of the AZ61A alloy and at one million cycles for the AZ31A alloy. Both the magnesium alloys have been found to be damaged in fatigue by understressing as well as by overstressing, whenever the prestress condition was above the damage line. On the other hand, neither the fatigue strength nor the fatigue life of either of these alloys was raised, or extended, by understressing below the damage line.

In either case, the prestress damaging effect was greater as the prestress increased, indicating the damage to be dependent more upon the magnitude of the prestress rather than upon the number of cycles.

The tensile properties of the magnesium alloys seem to be unaffected by prestressing occurring either above or below the damage line.

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DISCUSSION

MR. P. R. TOOLIN.[1]—As the fatigue machines used in this investigation were of the constant deflection type and as the stiffnesses of specimens from some materials change appreciably during a fatigue test, one wonders if the authors noted any such change in these magnesium alloy specimens.

In regard to the damage line, it would be interesting to know if an attempt was made to associate this line with very minute cracks or with large amounts of slip. Observations on the rate of growth of the fatigue cracks would also be of interest.

MR. EVAN H. SCHUETTE.[2]—The authors describe a part of the effect of damage as a "reduction in fatigue strength." If the existence of an endurance limit has been demonstrated, and if it is possible to prestress in some fashion so that the subsequent endurance limit is clearly lowered, then it can truly be said that a reduction in fatigue strength has occurred. In the present instance, however, there is no evidence that the endurance limit has been reached. Thus the movement of the *S-N* curve might better be referred to as a reduction in fatigue life.

With reference to Mr. Toolin's comments, we have made some preliminary investigations of the crack-propagating characteristics of two sand-cast magnesium alloys, H-T4 and ZK51A-T5. The H-T4 gave extrapolated evidence that cracking began almost from the initial cycle, propagating slowly and steadily thereafter—an especially convenient situation from the inspection standpoint. In ZK51A-T5, however, no evidence of cracking was found until just prior to complete failure. While these results are from a rather sketchy investigation, they do indicate the possibility of wide variation among alloys, along with the implication that initial cracking may in some cases have doubtful significance as a measure of fatigue life.

We have found that the errors resulting from work stiffening of a specimen during a constant-deflection fatigue test are quite small for hard-rolled material, such as J-1h* or FS-1h.* Changes of this kind are of real significance when testing material that is initially in a relatively soft condition.

MR. FRANK A. MCCLINTOCK.[3]—What results are obtained when the effect of overstressing is analyzed according to Miner[4] by summing for each specimen the number of cycles at each stress divided by the number of cycles to failure of a virgin specimen at that stress and comparing the sum to unity?

MESSRS. J. VIGLIONE AND F. S. WILLIAMS (*authors' closure*).—The authors wish to thank Messrs. Toolin, Schuette, and McClintock for their discussion of this paper. In reply to Mr. Toolin's questions, no change in the stiffness of the J1-

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[1] Mechanics Department, Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] The Dow Chemical Co., Midland, Mich.

* Current designations: J1-H24 and FS1-24.

[3] Assistant Professor, Massachusetts Institute of Technology, Cambridge, Mass.

H24 and FS1-H24 magnesium alloys test specimens was found to occur during the progress of the tests. With regard to the damage line, no attempt was made to relate the formation of minute fatigue cracks with the location of the line. However, in the use of specimens of these alloys to obtain small fatigue cracks for sensitivity studies of dye inspection penetrants it was found that the cracks formed late and progressed very rapidly.

Mr. Schuette has raised an interesting point in connection with the description of the damage, in holding that the damage is better expressed as a reduction in fatigue life rather than a reduction in fatigue strength. In view of the fact that an endurance limit has not been reached in this case, it appears that either of the two expressions describes the damage, that is, a reduction in fatigue life would shift the S-N curves to the left and in the process result in the fatigue strength (stress for a specified finite life) being reduced. Mr. Schuette's observations regarding the smallness of errors resulting from work stiffening of these alloys during constant-deflection fatigue tests are appreciated.

In reply to Mr. McClintock's question, no attempt was made to analyze the data according to Miner.[4]

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[4] M. A. Miner, "Cumulative Damage in Fatigue,' *Journal Applied Mechanics*, Vol. 12, Sept., 1945.

AN INVESTIGATION OF THE PROT ACCELERATED FATIGUE TEST*

By E. J. Ward,[1] R. T. Schwartz,[1] and D. C. Schwartz[1]

Prot (1)[2] has proposed a method of accelerated fatigue testing for the purpose of obtaining the endurance limit in less time than is required by determining the usual stress-cycle diagram. The latter procedure is particularly time consuming if it is desired to obtain an estimate of the scatter about the endurance limit, because tests of a large number of specimens for a large number of cycles of stress are required.

The Prot procedure consists of continuously increasing, at a constant rate, the amplitude of the cyclic stress in a specimen, starting at a low stress below the estimated endurance limit, until failure occurs. Specimens are tested at several rates of increase of the amplitude, and the failure stresses obtained are plotted against the square root of the rate. The cyclic stress frequency in all cases is constant. Prot proposed that a straight line drawn through the failure stresses will intersect the zero rate at the endurance limit.

This work was undertaken as a preliminary investigation of the accuracy of Prot's accelerated method for estimating the mean endurance limit, and also the scatter about the endurance limit, for virgin and for welded SAE 4340 steel.

MATERIAL

Rotating-beam specimens were machined from the shanks of two propeller blades, referred to as blades 28 and 37. Two groups of specimens were taken from each blade; in one group the specimens were parent metal and in the other group the specimens contained a transverse flash weld at the center. The material was SAE 4340 steel heat treated to a Vickers hardness of 360 (estimated tensile strength of approximately 160,000 psi).

PROCEDURE

All specimens were tested in R. R. Moore Type rotating-beam machines. The specimens were the standard R. R. Moore type with a 5-in. radius and a minimum diameter of 0.300 in. The machines on which all the tests were run, except those tested at the fastest accelerated rate of loading, operated at 10,600 rpm. Those used for the fastest rate of loading operated at 3450 rpm. It is known that this difference in speed of cyclic loading does not appreciably affect the fatigue strength in normal fatigue testing (2).

Three rates of increase of the cyclic stress amplitude, which Prot terms "progressive loading," were used. They were accomplished by increasing the stress 3000 psi each 300,000 cycles, 3000 psi

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Wright Air Development Center, Dayton, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 891.

each 75,000 cycles and 3000 psi each 33,333 cycles. These increments resulted in average rates of loading of 0.01, 0.04, and 0.09 psi per cycle, respectively. It was assumed that an increase by increments would not seriously differ from a continuous increasing of the amplitude of cyclic stressing.

The stress was increased by manually adding bags of shot weighing 3.75 lb to the machine weigh pan. Since the diameter of the specimens varied a few thousandths of an inch, it was necessary to vary slightly from the number of cycles previously listed in order to maintain the same loading rate for all specimens. The time to failure was 4 to 6 hr for the 0.01 rate and 1 to 3 hr for the other two rates.

TABLE I.—HARDNESS DATA.

Specimen	Stress, psi	Cycles	Fracture	Average Vickers Hardness, 2½-kg Load		
				Parent	Metal[a]	Weld
WELDED SPECIMENS						
28W-23	78 200	495 000	Half through, half outside weld	365	365	359
37W-20	77 800	1 128 000	Through weld	371	315	312
28W-22	83 140	1 217 000	Outside of weld	363	341	348
37W-5	94 800	682 000	Outside of weld	350	376	383
PARENT METAL SPECIMENS						
28N-17	75 000	2 560 000	...	363	356	...

[a] The two values are from the two halves of the specimen.

In order to determine if Prot's method can be used to estimate the scatter about the endurance limit, a number of specimens were tested at each rate of loading used. For comparison, it was necessary to determine this scatter about the endurance limit.

One method of determining scatter about the endurance limit is to test a specimen at a certain stress, and if it has not failed after 10^7 cycles (for steel) another specimen is tested at a slightly higher stress. If the second specimen does not fail after 10^7 cycles another one is tested, and this is continued until a specimen fails. The whole procedure must be repeated enough times to establish the scatter. In this investigation, the procedure used differed from this only in that, if a specimen ran 10^7 cycles without failure, testing was continued at the next higher stress until failure occurred. The increments used were 5000 psi, and the failure stresses were taken as the endurance limits; this means the endurance limits actually could be up to 5000 psi lower than these failure stresses. With this procedure the question of coaxing arises. It may be noted that studies by the University of Illinois (3) indicate that the degree to which the endurance limit may be improved by coaxing depends upon its capacity for strain aging. The fatigue strength of non-ferrous metals and high hardness steels shows little or no increase due to coaxing. Therefore, it is believed that the procedure used gave results very close to the actual endurance limit, and this is confirmed by the fact that the results check the available rotating-beam endurance lim't data for SAE 4340 and other steels of this hardness.

Because of the use of the same specimen at different stresses, this procedure actually becomes a very slow loading rate of Prot's method, (although it is not continuous but in 10^7 cycle steps), and it is of course not an accelerated test. Therefore, in plotting the results of the accelerated tests to compare with the endurance limit, the comparison is not made at the zero ordinate, but at a rate of 0.0005 psi per cycle, even though there is only a very slight difference between the two.

TABLE II.—FAILURE STRESSES OF VIRGIN SPECIMENS FROM BLADE 37N.

Specimen	Loading Rate, psi per cycle	Stress^a at Failure , psi
FATIGUE TESTS FOR ENDURANCE LIMIT DETERMINATION		
No. 37N-1		95 000
No. 37N-3		80 000
No. 37N-6		85 000
No. 37N-8		90 000
No. 37N-10		80 000
No. 37N-13		95 000
No. 37N-21A		85 000
ACCELERATED FATIGUE TESTS		
No. 37N-4	0.01	89 000
No. 37N-19		93 000
No. 37N-22		101 000
No. 37N-23		89 000
No. 37N-24		86 000
No. 37N-27		92 000
No. 37N-28		90 000
No. 37N-2	0.04	101 000
No. 37N-9		102 000
No. 37N-11		95 000
No. 37N-12		95 000
No. 37N-17		99 000
No. 37N-18		101 000
No. 37N-26		101 000
No. 37N-5	0.09	105 000
No. 37N-14		99 000
No. 37N-15		118 000
No. 37N-16		107 000
No. 37N-20		105 000
No. 37N-21		111 000
No. 37N-25		110 000

[a] Initial stress 65,000 psi in all cases.

The tests for the determination of the endurance limit were run first. The welded specimens were initially stressed at 35,000 psi. The cyclic stressing on the specimens for the determination of the endurance limit was started in the late afternoon, so that 10^7 cycles were completed by the next morning, when the machine was stopped. That afternoon the cyclic stressing was resumed at an increase in stress of 5000 psi. In this manner 10^7 cycles were accumulated every day until failure occurred. The lowest stress at which failure occurred was 45,000 psi, so that the welded specimens tested at the accelerated rates of loading were also initially stressed to 35,000 psi. The lowest stress at which a parent metal specimen failed was 75,000 psi, so the parent metal specimens tested at the accelerated rates were initially stressed to 65,000 psi. The procedure followed for the parent metal specimens was the same as that for the welded specimens.

TABLE III.—FAILURE STRESSES OF VIRGIN SPECIMENS FROM BLADE 28.

Specimen	Loading Rate, psi per cycle	Stress^a at Failure, psi
FATIGUE TESTS FOR ENDURANCE LIMIT DETERMINATION		
No. 28N-2		90 000
No. 28N-9		90 000
No. 28N-11		90 000
No. 28N-12		80 000
No. 28N-17		75 000
No. 28N-18		75 000
No. 28N-26		95 000
ACCELERATED FATIGUE TESTS		
No. 28N-5	0.01	95 000
No. 28N-14		104 000
No. 28N-15		92 000
No. 28N-16		98 000
No. 28N-20		84 000
No. 28N-21		83 000
No. 28N-25		86 000
No. 28N-1	0.04	104 000
No. 28N-3		96 000
No. 28N-6		101 000
No. 28N-7		104 000
No. 28N-8		104,000
No. 28N-10		95 000
No. 28N-13		111 000
No. 28N-4	0.09	108 000
No. 28N-19		98 000
No. 28N-22		110 000
No. 28N-23		110 000
No. 28N-24		107 000
No. 28N-27		110 000
No. 28N-28		113 000

[a] Initial stress 65,000 psi in all cases.

Vickers hardness measurements were made on four welded and one plain specimen using a $2\frac{1}{2}$-kg load (Table I).

RESULTS AND DISCUSSIONS

The failure stresses for all specimens tested are given in Tables II, III, IV, and V. A statistical analysis of the variance of the variables involved in the tests was made. The analysis did not reveal any significant difference between the results obtained from the two propeller blades; therefore, the data from the two blades were combined for plotting and calculations.

TABLE IV.—FAILURE STRESSES OF WELDED SPECIMENS FROM BLADE 37.

Specimen	Loading Rate, psi per cycle	Stress[a] at Failure, psi
FATIGUE TEST FOR ENDURANCE LIMIT DETERMINATION		
No. 37W-6		65 000
No. 37W-7		80 000
No. 37W-9		85 000
No. 37W-11		45 000
No. 37W-19		70 000
No. 37W-22		75 000
No. 37W-27		80 000
ACCELERATED FATIGUE TESTS		
No. 37W-3	0.01	77 000
No. 37W-4		80 000
No. 37W-8		83 000
No. 37W-10		83 000
No. 37W-12		80 000
No. 37W-14		81 000
No. 37W-23		77 000
No. 37W-2	0.04	86 000
No. 37W-16		83 000
No. 37W-20		78 000
No. 37W-24		62 000
No. 37W-26		80 000
No. 37W-5	0.04	95 000
No. 37W-13		83 000
No. 37W-15		80 000
No. 37W-17		90 000
No. 37W-18		87 000
No. 37W-21		98 000
No. 37W-28		89 000

[a] Initial stress, 35,000 psi in all cases.

From the data in the top portions of Tables II, III, IV, and V, the mean endurance limit for the parent metal specimens determined by the described procedure was 86,000 psi, and the mean endurance limit for the welded specimens similarly determined was 70,000 psi.

In making the statistical analysis of the data, it was assumed that the scatter in the failure stresses at each rate of loading was normally distributed. For each of the three accelerated rates of loading the mean failure stress and a standard deviation were calculated. In Fig. 1, the failure stresses for the plain specimens were plotted against the square roots of the rates of loading. The mean failure stresses were also plotted. A straight line fits the latter points very well, and intersects the 0.0005 psi per cycle ordinate at 86,000 psi, which is the mean endurance limit previously obtained. In Fig. 2, the same procedure was followed for the welded specimens, and the straight line drawn through the three mean failure stresses intersected the 0.0005 psi per cycle ordinate at 75,000 psi, compared to the mean endurance limit previously obtained of 70,000 psi. Therefore, the Prot procedure resulted in a better estimate for the parent metal specimens than for the welded specimen.

TABLE V.—FAILURE STRESSES OF WELDED SPECIMENS FROM BLADE 28.

Specimen	Loading Rate, psi per cycle	Stress[a] at Failure, psi
FATIGUE TESTS FOR ENDURANCE LIMIT DETERMINATION		
No. 28W-2		70 000
No. 28W-16		70 000
No. 28W-20		45 000
No. 28W-24		75 000
No. 28W-25		70 000
No. 28W-26		75 000
ACCELERATED FATIGUE TESTS		
No. 28W-5	0.01	86 000
No. 28W-13		75 000
No. 28W-15		78 000
No. 28W-17		75 000
No. 28W-18		65 000
No. 28W-21		81 000
No. 28W-28		78 000
No. 28W-6	0.04	88 000
No. 28W-7		84 000
No. 28W-9		89 000
No. 28W-11		78 000
No. 28W-19		77 000
No. 28W-22		83 000
No. 28W-27		89 000
No. 28W-1	0.09	95 000
No. 28W-3		89 000
No. 28W-4		74 000
No. 28W-10		87 000
No. 28W-12		89 000
No. 28W-14		80 000
No. 28W-23		78 000

[a] Initial stress 35,000 psi in all cases.

The 95 per cent confidence limits,

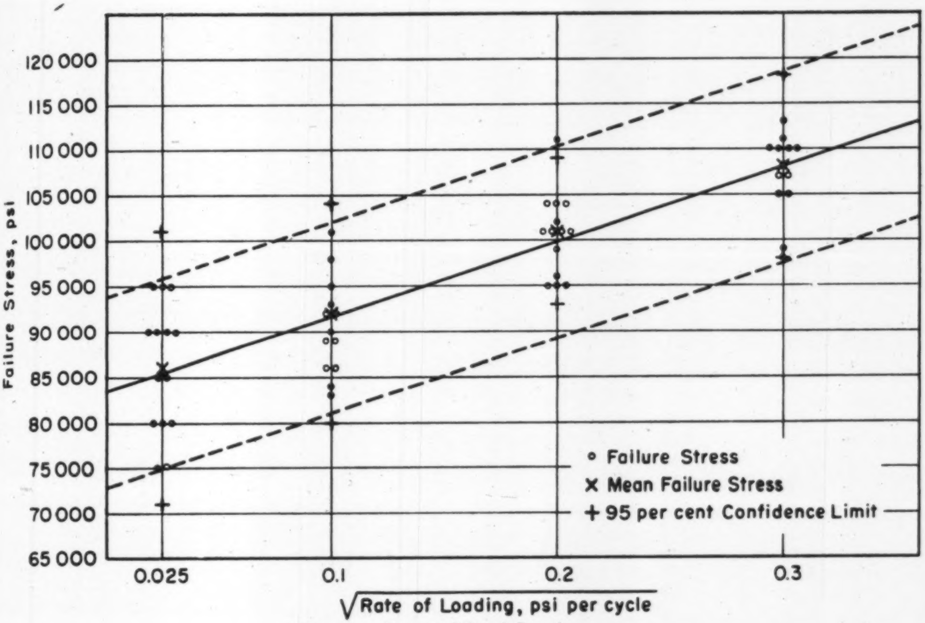

FIG. 1.—Parent Metal Specimens.

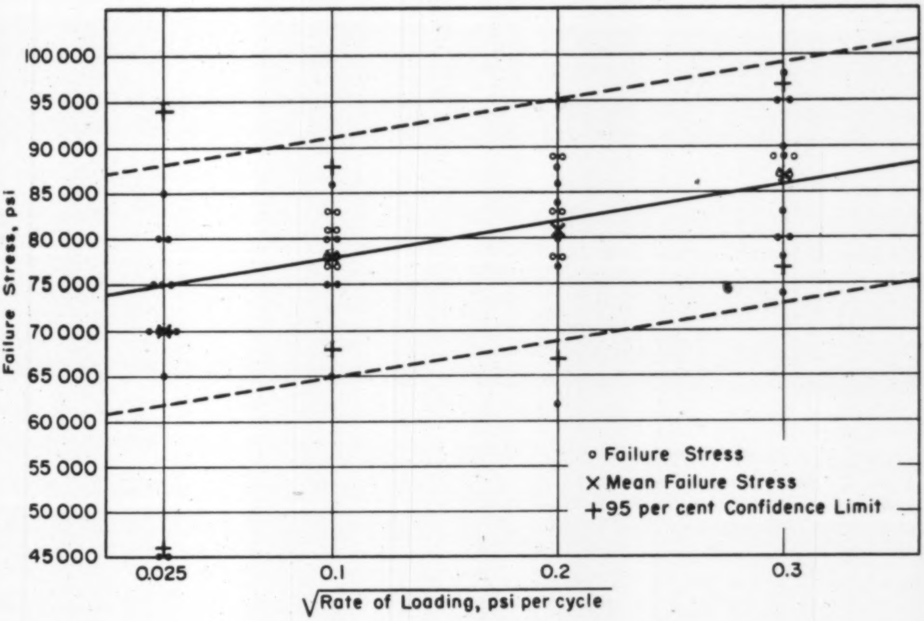

FIG. 2.—Welded Specimens.

which are the range of failure stresses within which it can be expected that 95 per cent of all specimens tested will fail, are equal to the mean failure stress ± twice the standard deviation and are shown plotted in Figs. 1 and 2. Since for the three accelerated loading rates there was not a significant difference in the scatter, the values of the 95 per cent confidence limits were averaged for these three loading rates, and the average was used to locate straight lines parallel to that through the mean values. Assuming these lines to give the ranges of the 95 per cent confidence limits at the 0.0005 psi per cycle ordinate, as seen in Fig. 1 for the parent metal specimens, the range obtained was 75,000 to 96,000 psi. All 14 of the check specimens had failed within this range. Also, the 95 per cent confidence limit range calculated from these 14 specimens was 71,000 to 101,000 psi. However, the range estimated by this procedure for the welded specimens was 62,000 to 88,000 psi, as seen in Fig. 2, and only 11 of the 13 check specimens failed within this range, while the other two failed at 45,000 psi. Also the 95 per cent confidence limit range calculated from these 13 specimens was 46,000 to 94,000 psi. Therefore a good estimate for the scatter about the mean endurance limit was obtained for the parent metal specimens, but not for the welded specimens. There is perhaps an explanation for this. It is believed that the four values falling outside of the confidence limits in Fig. 2, and possibly more, are members of a different population, probably defective welds. If such a second population exists among welded specimens, three major problems arise: (1) What is the probability of a welded specimen selected at random being a member of such a population? (2) What is the distribution function of this population? and (3) Will the mean failure stresses at each loading rate vary directly as the square roots of the loading rates? If the assumption of a second population among welded specimens is a correct one, the presence of extreme values at the lowest loading rate and the apparent absence of any such values at the highest loading rate may have three different explanations: (1) The mean failure stresses of the foreign population at each loading rate vary directly as the square root of the loading rates, but the regression equation has a higher slope than for the population of welded specimens as a whole. (2) The scatter in the second population is much larger at lower loading rates than at higher. (3) The mean failure stresses at each loading rate do not vary as the square roots of the loading rates in the second population.

Study of the failure stresses, magnaflux indications, position of fracture, and flow line pattern yields the following conclusions: (1) In general, specimens failing at stresses above 75,000 psi broke outside the weld and those failing at or below 75,000 psi broke through the weld. (2) Specimens failing at unusually low stresses had magnaflux indications of flaws and exhibited a tendency toward a dendritic structure. These support the theory of a second population in the scatter of the welded specimen results.

After this work was completed, other results (4) have been reported in this country on investigating Prot's procedure. These investigations found that the Prot method gave results for ingot iron which compared favorably with the ordinary methods of fatigue testing, while results for 75S-T aluminum alloy did not compare favorably.

Additional investigation, particularly involving larger number of specimens if scatter is to be considered statistically may modify these conclusions. The effect of varying the initial stress employed in the Prot procedure should be included in further investigation, although it is believed that this feature is probably not of major influence provided the initial stress is well below the endurance limit. It is also desired to point out that investigations to check the applicability of the Prot procedure will necessarily involve more time and tests than would be involved in the use of the method after it has been confirmed and established for certain materials. For example, if there is confidence that the procedure is applicable for a given material and condition and if the slope of the failure stress - square root of loading rate line, with scatter band, has been established tests could conceivably be made at only one loading rate to predict the endurance limit. This would be especially advantageous in saving time and effort in certain cases where it is desired to run fatigue tests for control purposes.

CONCLUSIONS

Based on the limited results of this investigation, a good estimate of the endurance limit and the scatter about the endurance limit for SAE 4340 steel was obtained by utilizing Prot's procedure. Prot's procedure gave a fair estimate of the endurance limit, but not of the scatter about the endurance limit, for welded SAE 4340 steel. This is believed to be a result of defective welds forming a second population, as illustrated in Fig. 2, by the four values falling outside the confidence limits. Perhaps if a very large number of specimens were tested, the second population could be defined well enough to permit an accurate estimate of the scatter. The results would appear to justify recommending further investigation of Prot's method.

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(3) University of Illinois in cooperation with the Office of Naval Research, "An Investigation of the Behavior of Materials Under Repeated Stress," *Status Report*, Dec., 1951.

(4) A. P. Boresi and T. J. Dolan, "An Appraisal of the Prot Method of Fatigue Testing (Part 1)," *Technical Report No. 34*, Contract N6-Dri-71, T.O. IV; Project NR-031-005, University of Illinois, Jan., 1953.

DISCUSSION

Mr. T. J. Dolan.[1]—We have been making an extensive study of the Prot method in the Mechanics Department at Illinois; it might be of interest to discuss some of the concepts we have on the adequacy and utility of the method.

Fundamentally it is an efficient method in that every specimen tested is averaged or weighted in determining the final endurance limit. When employing a conventional *S-N* curve, the endurance limit is determined primarily by the few specimens that are tested at the lowest stress levels; in the Prot procedure every specimen is included in the statistical summary made to determine the most probable endurance limit.

Prot in his original thinking proposed the method as a rapid method that would allow determination of the fatigue limit with a limited number of specimens and with very short lifetimes of testing. For example, determination of the fatigue strength of non-ferrous metals for 500 million cycles of stress is time consuming; Prot suggested that his method would only require the testing of specimens at relatively short lifetimes and still predict about the same endurance limit.

There is nothing sacred about the conventional *S-N* curve. Possibly if Prot had suggested his method and it had been generally adopted before the conventional *S-N* curve was proposed, we might all be wondering whether the *S-N* curve was adequate to express the fatigue resistance of a metal.

I should like to inquire as to the exact method of analysis of the final data in the authors' paper. For example, at each value of loading rate the authors tested a number of specimens; what method was used to determine the confidence limits at this loading rate and to extrapolate to the ordinate at zero loading rate?

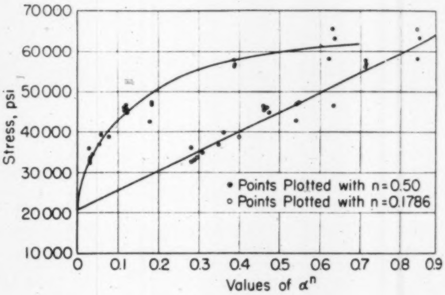

Fig. 3.—Appraisal of Prot Method for 75S-T Aluminum Alloy.

In order to make the Prot procedure applicable to small groups of samples, it would be desirable to be able to test (let us say) only one specimen at each of four or five loading rates, and then analyze the data to determine the most probable endurance limit. This would require a different analysis in the statistical approach which we are trying at the present time. If the fracture stress is plotted *versus* the loading rate α with an exponent of one half as in the authors' paper, one finds that for some metals the data do not fall along a straight line. However, by using the proper exponent n, a straight line can be obtained. For example, in the accompanying Fig. 3 for an aluminum alloy, a pronounced curve is obtained for $n = \frac{1}{2}$, and the data cannot be readily extrapolated back to $\alpha = 0$. With $n = 0.1786$, the same data group along a straight line that can be extrapolated back to determine the Prot endurance limit. Mathematically one can analyze the data to determine the optimum value of n to which to plot the data, and then determine the best straight line by the principle of squares. We are studying these procedures with methods for evaluating the relative standard deviations of the data and how the deviations may be affected by extrapolation.

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[1] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

For example, if the least value of loading rate α is large, the value for endurance limit includes the uncertainties of a large extrapolation. The question arises as to whether the standard deviation at the origin (that is, for $\alpha = 0$) is the same as for the group of experimental data at any selected value of α. There is no fundamental reason to suspect that the standard deviation at each loading rate should be the same. By combining these factors mathematically we hope to be able to predict from the data the most probable endurance limit and its standard deviation.

The question of coaxing always comes up with tests of this nature, and as part of our experimental program, we are testing low- and high-carbon steels and aluminum alloys. It appears that the values we are obtaining for predicted endurance limit for low-carbon steels are only slightly larger than those determined by the ordinary method. Perhaps the process of extrapolation tends to bring the final value back to about the same endurance limit for those low-carbon steels which normally coax quite a bit as that obtained by the conventional method of employing *S-N* curves.

The Prot method will determine an endurance limit for non-ferrous metals which *do not* have definite endurance limits; however, this value might be visualized as a sort of lower asymptote to the *S-N* curve for those metals which do not have a sharp, abrupt break in the curve.

The Prot test should be useful as a convenient manufacturing check on processing operations where a change in manufacturing processing might affect the endurance limit. By testing a few specimens under this procedure one could rather quickly obtain a relative index of whether the endurance limit were adversely affected or improved.

MR. B. J. LAZAN.[2]—I should like to amplify somewhat Mr. Dolan's comments regarding the variability of Prot exponent n. We have been doing some progressive load increase work on aluminum and like Mr. Dolan find that an exponent of approximately 0.18 fits the test data much better than 0.5. Similarly for the case of mild steel an exponent n different from 0.5 produces the line of greatest straightness. The choice of exponent in both cases greatly affected the extrapolation process and the resultant value determined for the fatigue limit.

A second point that should be amplified in connection with progressive load increase testing is the effect of understressing or coaxing. In recent tests by Sinclair[3] a stepwise load increase method was used on materials which are strain ageable and others which are not. Although Sinclair did not include Prot method consideration in his work, his data are replotted in the accompanying Fig. 4 to show failure stress *versus* average loading rate during the step load increase. This figure indicates an agreement with Prot's assumption for 75S-T6 aluminum alloy and 70-30 brass which are not suceptible to strain aging according to Sinclair. However, materials such as 2340 and 1045 steel, which are susceptible to strain aging, display such large coaxing effect as to cause a reverse slope to the Prot curve; thus the Prot method is misleading.

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[2] Professor of Materials Engineering, Univresity of Minnesota, Minneapolis, Minn.

[3] G. M. Sinclair, "An Investigation of the Coaxing Effect in Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 743 (1952).

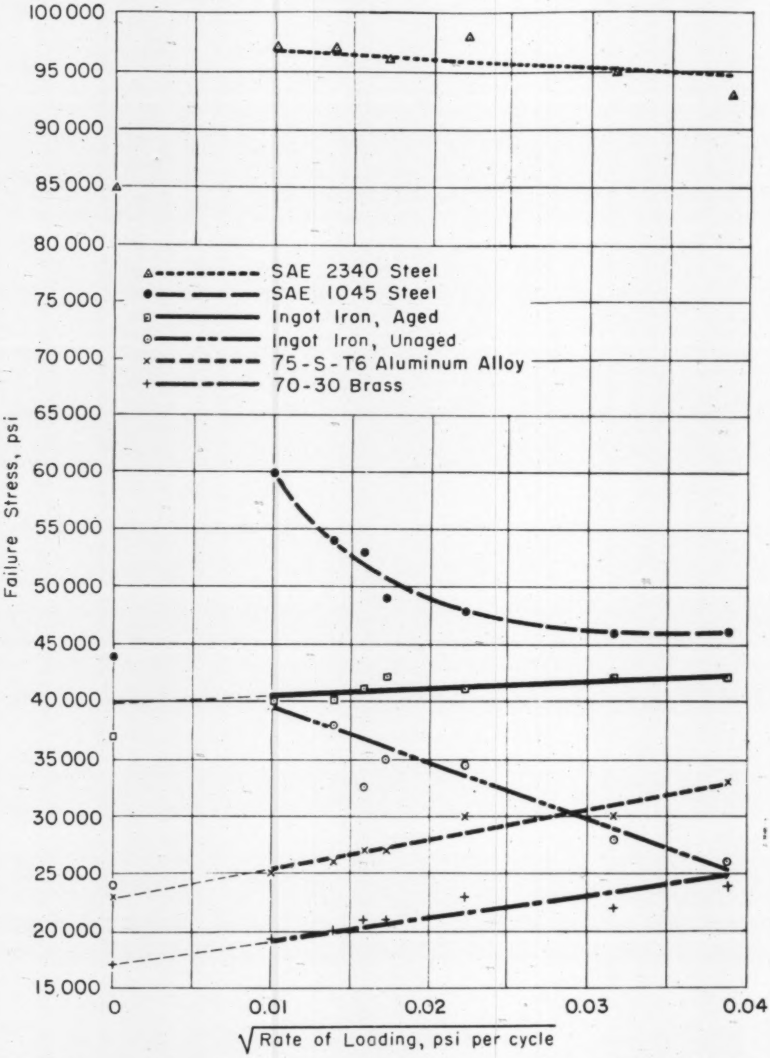

FIG. 4.—Failure Stress *versus* Square Root of Loading Rate of Various Materials. Stepwise Load Increase.

In conclusion, this progressive load increase method should be used with caution. Even though this method does seem to be reliable for certain classes of material and may be a good comparative test for industrial inspection, nevertheless in applying it to new materials it should be first carefully checked against the constant load fatigue method.

MR. PAUL KUHN.[4]—The authors mentioned only very incidentally something which has come up in other papers, namely, that weld defects add another population. I wonder if that is an adequate description of the status or whether we should not assume that weld defects add more nearly an infinity of populations which makes the problem quite a bit more difficult.

MR. E. J. WARD (*author*).—In regard to Mr. Dolan's comments, the question of the same standard deviation at the different rates is something that we wondered about, and the only thing that we could do was to check the three rates that we had used and the statisticians could find no significant difference between the standard deviations or the amount of scatter at the three rates that we used. Whether this would always be true or not I could not say.

There is one place that I would like to differ with Mr. Dolan, and that is the statement that some metals do not have endurance limits. I believe that from our experience and from what information we have been able to get from other people that all metals do have endurance limits. Now it is very true that some of them may continue to drop far beyond the 10 million cycles we usually use for steel, but the first information that we obtained in our own laboratory on that was several years ago. Three specimens of Alloy 75 ST were run out to 7, 8, and 9 billion cycles without any failure. They were set at stresses just slightly below the endurance limit and after they had run out to that many cycles the stress was raised, they were retested at several thousand lb per sq in. above the endurance limit, and they fell to the high side of our *S-N* curve. Therefore I feel that until we get information to the contrary we cannot make the statement that metals do not have endurance limits. It is possible that the *S-N* curve theoretically continues to drop, but if so, it is believed that it is asymptotic to some value which would be the endurance limit. In other words it does not eventually drop to zero.

In regard to Mr. Kuhn's statement—I agree wholeheartedly that it is entirely possible that defective welds may produce an infinite number of populations—I certainly could not investigate that question. I think that would be a little too large a project to undertake.

I think Mr. Lazan's caution on the use of the Prot method is well taken, that we do find materials already which do not seem to give the accurate endurance limits but I think it can be used if used with caution.

The way we got into the Prot method is that our Propeller Laboratory at Wright Air Development Center was very interested in getting into the question of quality control of the fatigue limits of steel used in propeller blades, and the Prot method in this case would be a very good control if the initial work can be done to set up the slope of your line and predict the endurance limit. After that you need only test at one rate from then on. For each new lot of steel which you obtained you could run a number of specimens at an accelerated rate and check it against the line through the mean stresses.

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A RANDOM FATIGUE TESTING PROCEDURE AND MACHINE*

By Alfred M. Freudenthal[1]

Synopsis

A procedure is outlined for rotating-beam fatigue tests under a random sequence of load impulses derived from an assumed frequency distribution function of the expected loads, and a fatigue testing machine designed and built for the purpose of realizing such a testing procedure is described.

From an assumed exponential distribution function of the loads, which is known to represent fairly well maneuver and gust loads of airplanes in operation, four random sequences of load impulses, differing by the specified frequency of the highest load level, have been constructed. Twelve small specimens of 75S-T aluminum have been tested under each of these load sequences, the test results plotted on extreme value probability paper, and the cycle ratios, mean stresses, and equivalent constant stresses computed.

The test results indicate that the sum of the cycle ratios does not provide a means of even roughly estimating the cumulative damage under a varying level of repeated stress; only systematic tests under random sequences of load impulses derived from known distribution may provide the approach to a rational and effective study of the problem of cumulative damage in fatigue, and thus lead to the development of a criterion for such damage that could be used in design.

Fatigue Testing Under Varying Load Amplitudes

In conventional procedures of fatigue testing, the purpose of which is not to find the endurance limit but to determine the life as a function of certain testing conditions, a sequence of such conditions, defined either in terms of mean stress and stress amplitude or in terms of a stress range, is applied repeatedly and the number of repetitions or cycles to failure recorded as a function of the two characteristic stress levels. For full stress reversal (zero mean stress) one stress level alone specifies the testing condition; for tests under combined stress it is a certain invariant combination of the six components of the stress tensor, such as the octahedral stress or the stress intensity (**1**)[2] that fully describes this condition. Since the performance in service of structural and machine parts under the dynamic conditions of repeated loading or vibratory stress is usually associated with stress cycles of rapidly varying amplitude and more or less random sequence, the correlation between the results of conventional fatigue tests and the actual fatigue resistance in service is rather vague, even if the problems of the statistical reproduction of results and of the effects of size and of shape of the specimen on its fatigue performance (which make such correlation still more vague) are not considered.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 909.

Numerous attempts have been made to provide more meaningful results of fatigue tests either by running tests at a periodically repeated short sequence of stress cycles of varying amplitude (2, 3, 4, 5), or by *a priori* establishing so-called laws of cumulative damage according to which the damaging effects of stress cycles of different amplitudes can be superimposed and the fatigue life under given load sequences predicted from the results of conventional tests (6, 7, 8). Both approaches have so far failed to provide a rational procedure for the interpretation of the results of fatigue tests.

In the first the fatigue life depends significantly on the character of an arbitrarily selected, frequently repeated, short-load sequence—for instance, on the specific shape of the periodic change of the load amplitude and the length of the period (5) as well as on the fact whether the first load sequence starts with the peak load or with the minimum load (2); the meaning of the test results is, therefore, limited to the particular load sequence, which usually has no recognizable relation to the load sequences expected in service. In the second approach the simple relations between rate of damage and number of load cycles on which these "laws" are based (constant relation in Miner's cumulative damage concepts (6), exponential relation in Shanley's concept (8)) do not represent sufficiently close approximations of the real, rather complex, and probably not monotonic progress of damage (9).

Though Shanley's concept appears to be more realistic than the linear "law," its practical application is much more difficult, since the momentary damage rate at any stress level necessarily depends on the integral over the history of all preceding damage processes; the simple algebraic summation of damage effects on the various levels, which is the principal attractive feature of the linear law, is thus lost.

Recent investigations of fatigue life under specific conditions of overstress or understress (2, 3), as well as under periodic triangular (4), sinusoidal, or exponential (5) load sequences have neither provided test results that would be more generally interpretable and more meaningful in terms of service performance than are the results of conventional fatigue tests at constant load amplitudes, nor (as could have been expected *a priori*) have they been able to justify the rather widely used law of linear cumulative damage based on a constant damage rate characteristic of each stress level. It appears that the results of fatigue tests performed with load cycles of amplitudes varying according to a definite, usually periodic, program are as difficult to interpret in terms of performance under actual service conditions, and are as vaguely correlated with such performance, as are the conventional constant load amplitude fatigue tests. Since the progress of damage under the applied load program is a definite function of the particular program, it is as different from that of any other, sufficiently dissimilar program as it is from the progress of damage under a constant load amplitude.

A radically new approach to the programing of variable amplitude fatigue tests is therefore required if such tests are to provide sufficiently general and meaningful information concerning the fatigue performance of metals under actual service conditions; fatigue testing equipment must be designed in which such load programing can be easily and reliably realized. Such an approach is outlined and an experimental fatigue testing machine described that has been constructed in order to apply loading programs that can be made as representative of rationally specified service conditions as can reasonably be required. Some preliminary results obtained on this machine are discussed.

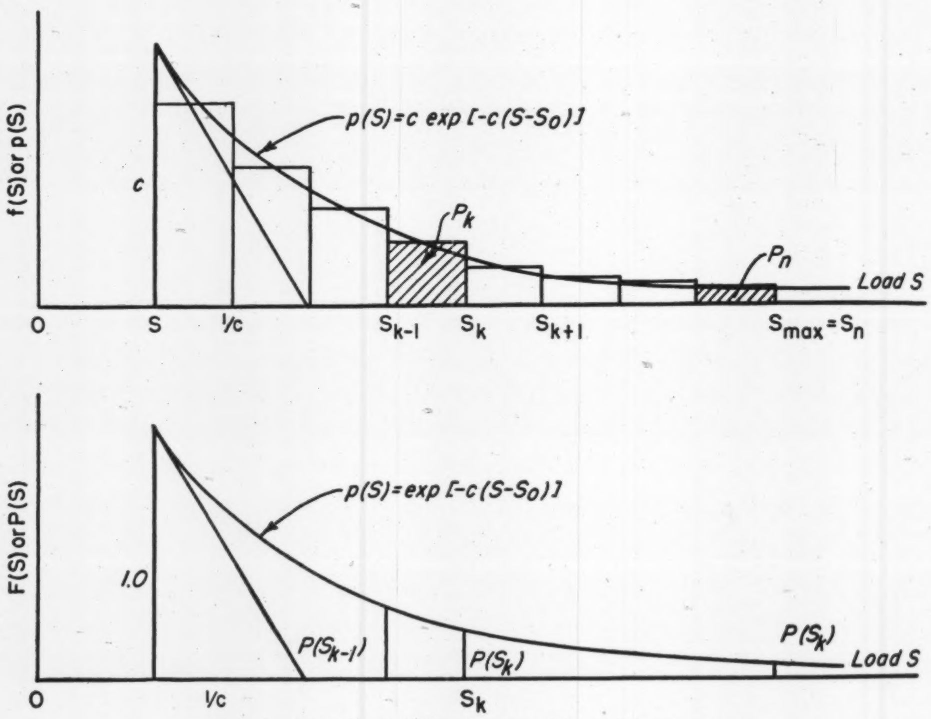

FIG. 1.—Exponential Distribution of Load Frequency.

STATISTICAL VARIATION OF SERVICE LOADING

Of the numerous types of engineering structures and machine parts the operating conditions of which make their fatigue performance significant, only aircraft structures have been subjected to systematic observations on the basis of which a rational estimate of the flight loads and of their frequency has become possible (10).

In a number of flight load investigations concerned with the statistical distribution of both gust loads and maneuver loads for various types of aircraft, relevant "load spectra" have been established indicating the relation between the load factor and its number of occurrence or its relative frequency. The analysis of these data has led to the conclusion that the relative frequency distributions $f(S)$ of both gust loads and maneuver loads in flight can, in first approximation, be represented by an exponential distribution (10):

$$f(S) = ce^{-c(S-S_0)} \qquad \text{..........(1)}$$

where c and S_0 denote the parameters of the distribution which are to be determined from the observations by a curve-fitting procedure, such as the classical method of least squares or the direct fitting by inspection of a straight line to the observation points plotted on semilogarithmic probability paper. S_0 represents the lower limit of the loads, while c measures the decrease of the frequency of the loads with the increase of their amplitudes. The exponential distribution which fits the maneuver loads better than the gust loads expresses the fact that the specific decrease of the load frequency $f(S)$ and therefore of its probability of occurrence $p(S)$ is proportional to the increase of the load amplitude, or

$$\frac{df(S)}{f(S)} \sim \frac{dp(S)}{p(S)} = -c dS \ldots\ldots (2)$$

The probability of a load amplitude higher than S is therefore

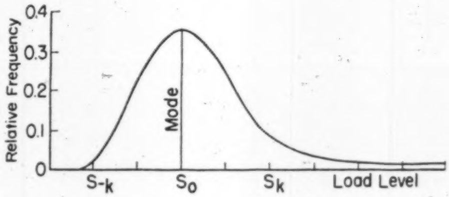

FIG. 2.—Extreme Value Distribution of Load Frequency.

$$P(S) = \int_S^\infty p(S)\, dS = \int_S^\infty ce^{-c(S-S_0)}\, dS = e^{-c(S-S_0)} \qquad (3)$$

and the probability of a load amplitude between the limits S_{k-1} and S_k is equal to

$$p(S_{K-1,\,k}) = P(S_{k-1}) - P(S_k) = p_k \ldots (4)$$

Equation 3 indicates that for a sufficiently large difference $(S_n - S_o)$ the probability $P(S_n)$ may become negligibly small; the distribution $p(S)$ which, theoretically, extends from S_o to infinity, may therefore with sufficient accuracy be replaced by the finite portion of $p(S)$ extending between S_o and S_n. If the segment $(S_n - S_o)$ is divided into n equal intervals the probability of occurrence of load amplitudes within the kth interval is determined by Eq 4. Replacing the varying load amplitudes within the intervals between S_{k-1} and S_k by the mean amplitude of this interval $S_{k-1,\,k}$, the continuous distribution function $p(S)$ may be represented approximately by n discrete probabilities of occurrence p_k of the n load amplitudes $S_{k-1,\,k}$ (Fig. 1).

If it is assumed that the statistical distribution of flight loads is fairly well described by Eq 1 so that their probability of occurrence can be predicted by Eq 4 for given parameters c and S_o, it is easy to devise a load program that can be made as representative of the flight loads as may be desired, by constructing on the basis of the discrete probabilities p_k a random time series of the load amplitudes $S_{k-1,\,k}$. For this construction either a numerical or an experimental procedure may be used; if the number n of load amplitudes is relatively small, the experimental procedure is rapid and sufficiently accurate.

A statistical population is made up of several hundred marbles of n different colors representing the n load amplitudes, so that the proportion of marbles of color k in this population is made equal to p_k. If marbles are now drawn at random and replaced and the sequence of colors is recorded, this record represents a random time series of the n load amplitudes. A fatigue specimen subjected to such a time series of load amplitudes may be assumed to sustain a load program as representative of actual service conditions as the exponential distributions of loads underlying it.

Each population from which the time series is constructed is characterized by the parameters S_o and c and by the scale factor of the load which determines the absolute value of the load intervals $(S_k - S_{k-1})$. Instead of specifying the coefficient c, which has no direct experimental interpretation, it is preferable to specify the probability p_n of the highest load amplitude $S_{n-1,\,n}$, from which the value of c can easily be determined. In this manner a group of random time series of load amplitudes can be constructed that differ in the specified frequency of occurrence of the most severe load amplitude. Since various groups may also differ in the absolute value of the load interval, conditions of different severity covering different ranges of stress can be reproduced.

It is evident that the random time series may be derived from any type of specified statistical population, not only from that based on the exponential frequency distribution Eq 1. This distribution has been used only as a particularly simple illustration of the procedure. Some load investigations have indicated that a skew distribution function of service loads with a mode not at but above the lowest load level (**13**), such as the distribution function of extreme values (Fig. 2), provides a closer approximation to certain operating conditions, particularly gust loads; such functions might therefore be used instead of the exponential distribution for the construction of the population.

RANDOM FATIGUE TESTING MACHINE

In order to impose a random time series of load amplitudes, a high-speed rotating-beam fatigue testing machine for small specimens has been designed and constructed. Figure 3 shows the main features of this machine, which operates on the principle of a conventional vertical rotating-beam machine with the added feature that the force acting on the cantilever specimen can be arbitrarily varied between zero and a maximum, so as to form a prescribed sequence of load and moment pulses of specified durations. The load sequence, after being constructed in the manner outlined, is recorded by punching a paper tape of such length that the effect of the periodicity of the load sequence, which is produced by the fact that the length of the recorded time series of load amplitudes and thus the length of the tape are necessarily finite, can be neglected. This requires a random sequence of at least several hundred, preferably a few thousand, load-impulses, and therefore a tape of considerable length.

When this tape is run through a reading device consisting of a group of contact springs, a group of relays operated by the small contact currents close a certain combination of circuits and deliver the prescribed sequence of current pulses through the windings of a vertically moving coil situated in the gap of a very strong permanent magnet (*1*). Since the force developed by the moving coil is proportional to the applied current, the variation of the current is transformed into a sequence of vertical force impulses. Careful calibration of the machine has shown that the current-force relation is practically linear over the operating range. The force impulses generated by the moving coil act upon a spring-suspended, 90-deg lever (*2*) which avoids all friction and backlash and exerts a horizontal force through a self-adjusting ball bearing (*5*) with spherical outer race on an extension arm of the specimen (*4*) connected to it by a clamping collet. Thus, the bending moment applied on the specimen (*3*) follows the force program punched on the tape.

The intensity of each of the n force pulses from the magnet can be varied by adjusting the current through the relays; the magnitude of the horizontal force exerted on the extension arm of the specimen in relation to the force from the magnet can be varied over a range from 1:1 to 4:1 by shifting the support of

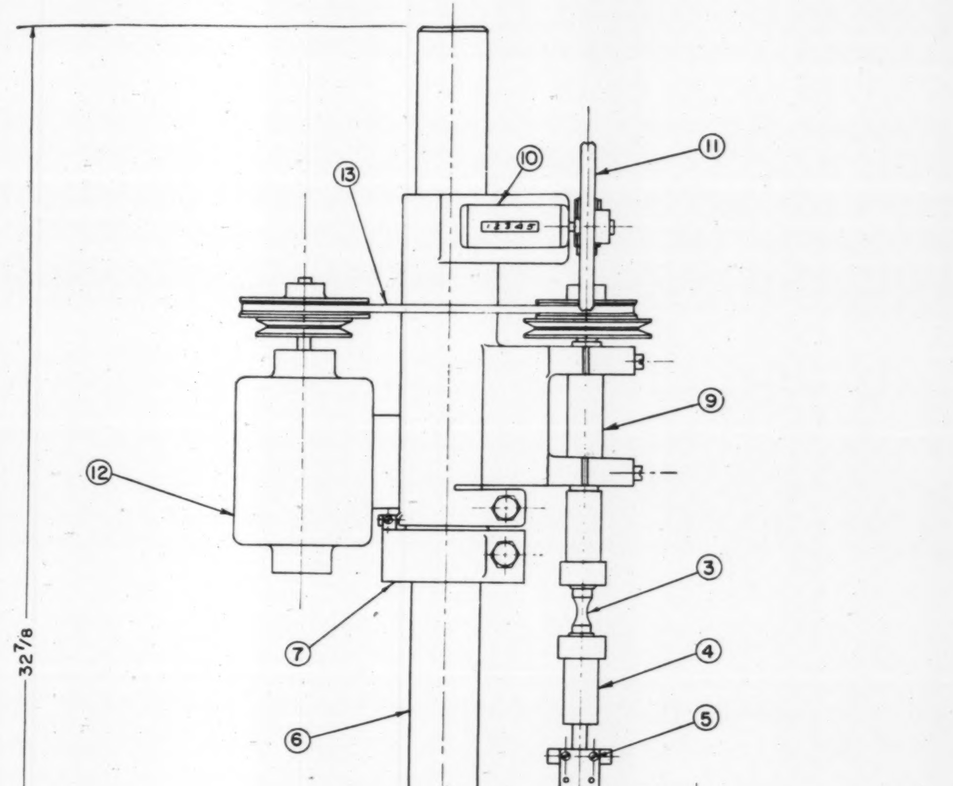
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FIG. 3.—Plan of Random Fatigue Testing Machine FRFM-1.

the ball bearing along the lever arm, while the bending moment changes with the changing distance between the critical section of the specimen and the point of horizontal force application on its extension arm. Since the maximum operating force developed by the moving coil will not exceed 10 lb, while the distance between the critical section of the specimen and the point of force application may vary roughly between 3.5 and 5.0 in., the bending moment at the critical section of the specimen might attain a maximum of 150 to 200 in.-lb, provided the deflection of the specimen under such a moment is compatible with the motion of the moving coil and lever.

The different elements of the machine are mounted on a rigid steel column (*6*) with the aid of brackets (*7*) in such a way that their relative position can easily be changed. The permanent magnet and the moving coil are placed in a brass container (*8*) and immersed in oil; thus, the heat developed in the coil is carried away and dissipated by the big surface of the oil container. This permits the use of relatively high currents and the application of the maximum vertical force of roughly 10 lb to the 90-deg lever. The specimen is gripped by two collets, one connected to the driving shaft (*9*), the other one to the center extension arm (*4*) which engages the spherical ball bearing referred to above. A contact spring, mounted on an insulating block near the main bearings of the drive shaft, is in steady contact with the shaft that is otherwise insulated from the rest of the machine, except the lever. During the test, a low current is run through the contact spring, the specimen, the lower collet, the spherical ball bearing and lever, and energizes a relay which controls the current of the driving motor. When the specimen breaks, this current is cut and the machine stops.

The main shaft is driven from a small motor (*12*) through a V-belt (*13*) and the speed is controlled by a Variac-transformer (*14*). A mechanical counter (*10*) driven from the main shaft by a worm gear (*11*) indicates the number of revolutions of the specimen.

The reading device for the punched tape has been designed for $n = 6$ force levels $S_{k-1,\,k}$ according to six lines on the tape along which holes are punched representing the random impulses on any of the six force levels. The tape can accommodate about 1000 punched holes and moves over a system of brass rollers, driven by a small motor.

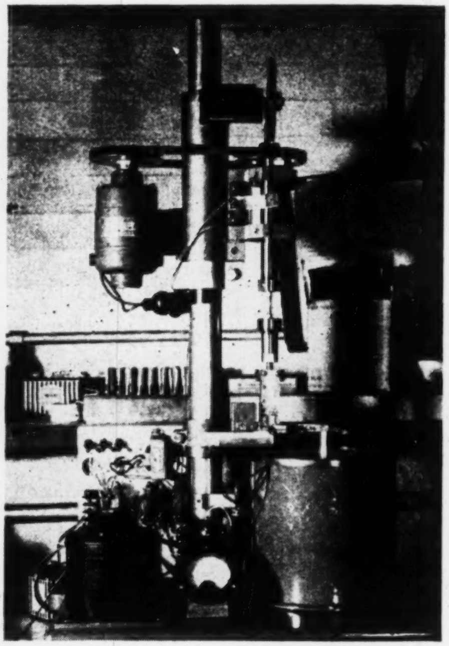

FIG. 4.—Random Fatigue Testing Machine FRFM-1.

As the punched holes pass the contact roller and springs, the relays on the various levels are operated, delivering the punched sequence of current pulses to the moving coil.

The traveling speed of the tape and the speed of rotation of the specimen can be adjusted within rather wide limits, so that the number of load cycles of constant amplitude applied during the passage of one punched hole under the contact spring, that is for each point of the prescribed load sequence, can be varied between 3 and 50. A slight overlapping of the punched holes prevents the load from going back to zero when changing from one level to another. During the short overlapping time, however, two relays are open; it has been found that during this time, which is a few per cent of the total impulse time, the actual force level is somewhere between the two levels, the actual position depending on the absolute value of their difference. Therefore, the change from one force level to another is not a perfect vertical jump, but a short and very steep transition.

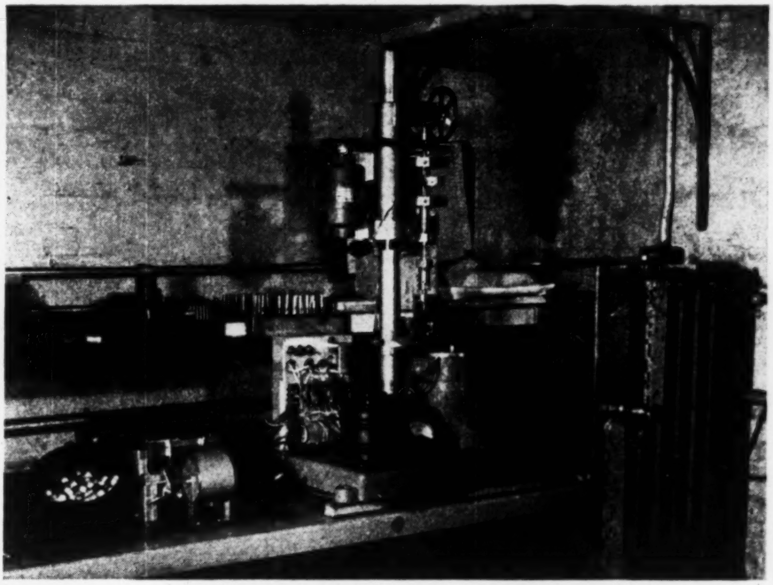

FIG. 5.—Fatigue Testing Machine with Punched-Tape Device.

Figure 4 is a photograph of the fatigue machine itself, which can be easily compared with the drawing in Fig. 3, in which the individual parts have been numbered. Figure 5 shows a photograph of the whole experimental apparatus including the tape-reading device with the punched tape and (on the left) the punching machine and the statistical population of loads made up of solid colored marbles.

RANDOM FATIGUE TESTS ON 75S-T ALUMINUM ALLOY

Probably even more than in conventional fatigue tests, the scatter of the test results must be considered as a feature of primary significance in random fatigue tests. Hence each test condition, specified by a random time series of load amplitudes, derived from a probability function of the load amplitudes, must be repeated frequently enough to permit a valid statistical interpretation of the results. For conventional fatigue tests the statistical distribution of extreme values has been suggested as the best approximation of the distribution of fatigue life to fracture under constant load amplitudes, with the logarithmic normal distribution as a close second

(**14, 15, 16**); a suitable probability paper has been developed on which fatigue test results can be plotted and, for non-ferrous metals, be fitted by a straight line (**16**). Nothing, however, is known concerning the distribution function of the results of random fatigue tests. In fact, it cannot even be assumed *a priori* that the distribution function of fatigue life under a random sequence of load amplitudes will have a sufficiently pronounced mode and a sufficiently narrow dispersion to make its statistical analysis of practical significance. It appears, therefore, that at least 25 repetitions of each test condition may be necessary to obtain results that can be plotted for comparison on various probability papers, with the purpose of finding the type of distribution function that is most likely to be associated with the results of random fatigue testing procedures.

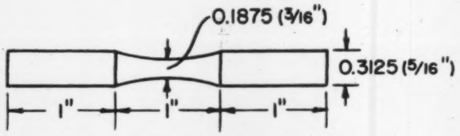

FIG. 6.—Rotating-Beam Specimen.

TABLE I.—RELATIVE FREQUENCIES OF OCCURRENCE OF SIX DIFFERENT LOAD LEVELS IN EXPONENTIAL DISTRIBUTIONS DEFINED BY p_6.

Load Level	Frequency	Distribution *A*	Distribution *B*	Distribution *C*	Distribution *D*
$S_{0,1}$........	$p_1 =$	0.56	0.43	0.36	0.25
$S_{1,2}$........	$p_2 =$	0.25	0.25	0.24	0.21
$S_{2,3}$........	$p_3 =$	0.11	0.15	0.16	0.17
$S_{3,4}$........	$p_4 =$	0.05	0.09	0.11	0.15
$S_{4,5}$........	$p_5 =$	0.02	0.05	0.08	0.12
$S_{5,6}$........	$p_6 =$	0.01	0.03	0.05	0.10

Since a rational interpretation of such results necessarily involves comparison between the results of random fatigue tests and results of conventional, constant-amplitude fatigue tests, the first random tests should be performed on a material for which statistically satisfactory data of conventional tests, obtained on similar and similarily polished specimens, are available, unless a large number of conventional tests were to be performed first to provide such data. The only structural metal for which adequate data have been published and statistically analyzed (**16, 17**) is 75S-T aluminum alloy. It was therefore decided to perform the pilot random fatigue tests on this alloy.

Rotating-beam fatigue specimens were machined to the dimensions shown in Fig. 6 and mechanically polished using successively finer grades of emery paper. The final polish was given with a 00 grade of emery and rouge and was done in such a way as to leave the scratches parallel to the axis of the specimen.

TABLE II.—MANEUVER LOAD SPECTRUM FOR FLIGHT TIME OF 100 HR OF MESSERSCHMIDT FIGHTER Me-109 DURING TACTICAL OPERATIONS.

Load Factor	Number of Cycles	Relative Load Frequency
1.4............	3200	0.427
2.2............	1800	0.240
3.0............	1100	0.147
3.8............	620	0.082
4.6............	350	0.047
5.4............	200	0.027
6.2............	120	0.016
7.0............	68	0.009
7.8............	39	0.005
Total number of cycles of $\Delta n \geq 1.4$..........	7497	1.000

Several exponential populations, made up of six load levels, were computed on the basis of assumed relative frequencies p_6 of the highest load level of 1, 3, 5, and 10 per cent. Table I shows the relative frequency distribution of the six load levels for the four assumed values of p_6.

It is interesting to compare the computed distributions with a maneuver-load spectrum for load factors $\Delta n \geqq 1.4$ derived from observations by Kaul (**10**) as modified by Wallgren (**4**) and reproduced in Table II. Considering the frequencies of load factors $\Delta n > 5.4$ as negligibly small, the frequencies of the six load factors $1.4 \leq \Delta n \leq 5.4$ show a very close agreement with the computed frequencies of the distribution *B* in Table I.

From each of the distributions presented in Table I a random time series was constructed and punched on a tape. In order to reduce the effect of the overlapping of the punched holes, a double hole was considered as a load unit, so that only about 500 terms of the time series could be punched on the tape length for which the roller system had been adjusted. At the test speed of 3600 rpm the number of constant stress cycles per load unit is roughly 10. Figure 7 shows characteristic parts of the time series *B* and *C*.

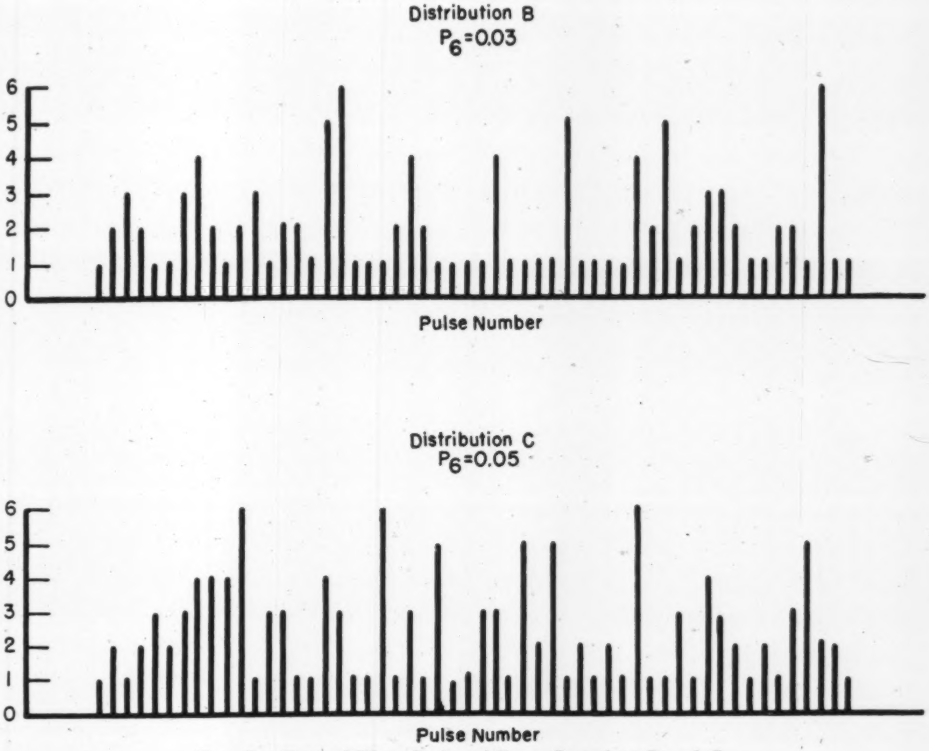

FIG. 7.—Part of Time Series of Force Impulses *B* and *C*.

The *S-N* diagram for small cantilever specimens of 75S-T as obtained by statistical evaluation of existing test results (**16, 17**) is presented in Fig. 8. The lowest load level $S_{0,1}$ was chosen so as to produce a moment causing a nominal maximum fiber stress in the critical section of the specimen of ±31,500 psi at which the expected (mean) life under constant stress level of specimens of the same size has been found to be of the order of magnitude of 5×10^6 cycles. The highest load level $S_{5,6}$ was selected so as to produce a moment causing a nominal maximum fiber stress of ±59,000 psi at which the expected (mean) life under constant stress level has been found to be between 10^4 and 10^5 cycles. The selected range of load levels thus completely covers the most significant portion of the *S-N* diagram. The uniform partition of this load interval produces a nominal stress-interval between the load levels of $[S_{k+1,\,k} - S_{k,\,k-1}] = 5500$ psi.

The nominal moments, stresses, and expected (mean) life under constant stress at the six load levels are given in Table III.

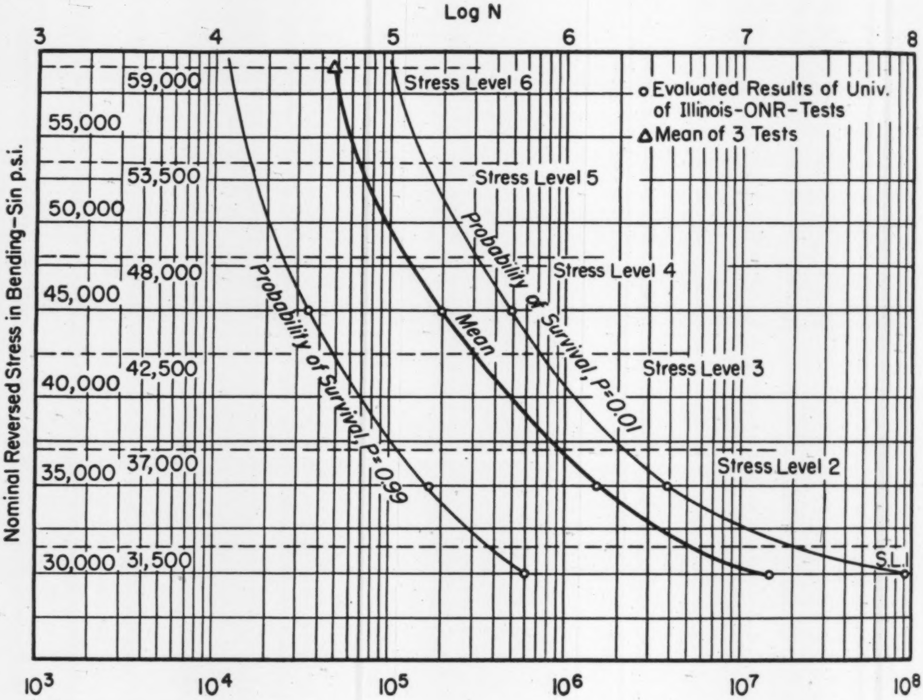

FIG. 8.—*S-N-P* Diagram for Small 75S-T Rotating-Beam Specimen.

The variation of the bending moment due to inaccuracy in the setting and operation of the resistances controlling the current and due to current fluctuation does not exceed ±1 per cent; the tolerance of the stress about its nominal value due to both moment variation and slight inaccuracies in the dimensions of the small specimens ($0.187 \leq d \leq 0.188$) does not exceed ±1.5 per cent. Considering that the selected stress interval is of the order of magnitude of 9 to 18 per cent of the applied stress levels, these tolerances seem reasonable, and can hardly be reduced for the very small specimens used in this investigation.

TABLE III.—CHARACTERISTICS OF APPLIED LOAD LEVELS.

Load Level	Moment, in-lb	Nominal Stress, psi	Expected Life
1......	20.25	±31 500	5×10^6
2......	23.85	±37 000	9×10^5
3......	27.45	±42 500	3×10^5
4......	31.05	±48 000	1.2×10^5
5......	34.65	±53 500	7×10^4
6......	38.25	±59 000	5×10^4

TEST RESULTS AND DISCUSSION

The preliminary results of the random fatigue tests are presented in ascending order in Table IV. Only 12 tests have been performed for each of the four time series derived from the exponential distributions *A*, *B*, *C*, and *D*. The results have been plotted as survivorship dia-

TABLE IV.—NUMBER OF CYCLES, *N*, TO FRACTURE, IN THOUSANDS.

Test, *m*	Distribution *A*	Distribution *B*	Distribution *C*	Distribution *D*
No. 1	438.8	84.9	52.6	59.9
No. 2	451.7	91.5	62.7	67.5
No. 3	467.5	112.8	66.0	74.7
No. 4	704.7	151.4	86.5	75.9
No. 5	725.2	156.8	92.7	85.8
No. 6	731.8	174.2	100.7	88.0
No. 7	986.8	179.6	103.1	100.0
No. 8	994.0	247.4	107.4	107.5
No. 9	1015.8	306.0	110.3	122.8
No. 10	1104.6	313.9	111.9	145.0
No. 11	1215.8	314.5	156.1	149.1
No. 12	1284.0	573.8	200.7	150.9
Mean life N[a]	~750.0	~175.0	~95.0	~93.0

[a] Estimated from Fig. 9.

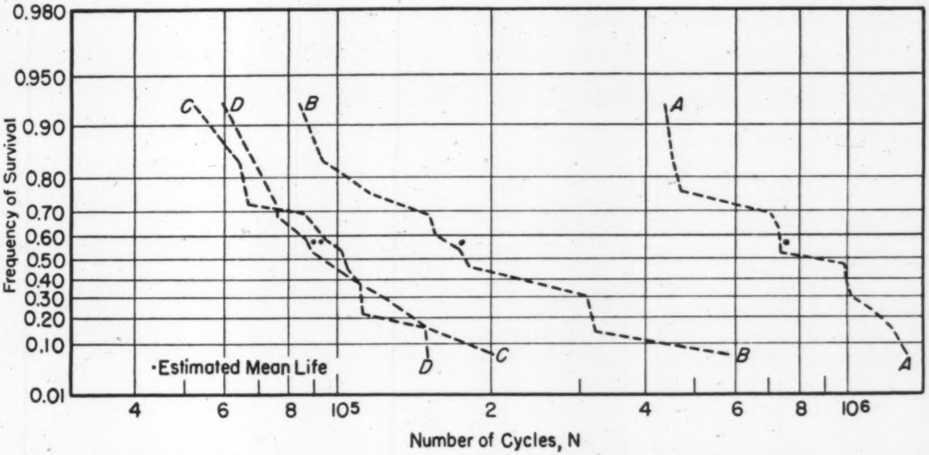

FIG. 9.—Survivorship Functions for Fatigue Tests Under Random Load Series *A*, *B*, *C*, and *D*

grams on extreme value probability paper (Fig. 9) with the individual plotting positions $F_m = 1 - \frac{m}{n+1}$, where m is the consecutive number of tests in one series (**13, 14**). The mean life $\bar{N}$ has been estimated from continuous survivorship functions fitted to the plotted diagrams by visual inspection.

The practical coincidence of the survivorship functions *C* and *D* is somewhat unexpected, and cannot be explained on the basis of our present simplified concepts of cumulative damage. A likely explanation would be to assume that the increased damage at the load levels 4, 5, and 6 of the time series *D* due to the higher values of p_k is compensated by decreased damage at the load levels 1, 2, and 3.

Another unexpected feature of the plotted survivorship functions is their somewhat reduced scatter in comparison to the results obtained from constant stress fatigue tests. While the scatter of those tests, as indicated by the slope of the extrapolated survivorship functions, ranges over 1 to 2 orders of magnitude between $P = 0.01$ and $P = 0.99$ at the constant stress levels of 30,000 to 45,000 psi (**16**), the scatter of the random fatigue test results extrapolated to the same probability limits appears narrower than one order of magnitude. Thus, in spite of the variable stress levels, the scatter is reduced. Whether this reduction is due to the influence of the high stress levels at which the constant stress scatter is, in general, significantly reduced because of the effect of the plastic deformation, or whether it represents a more or less statistical effect of the superposition of the distributions at the different load levels, similar to the operation of the central limit theorem in statistics, cannot be decided at present.

The cycle ratios $\Sigma n_k/N_k$ of the linear theory of cumulative damage for the four load distributions are easily determined by computing $\Sigma\ \dfrac{N \cdot p_k}{N_k}$ or, with $N = \bar{N}$, the mean cycle ratio $\bar{N} \cdot \Sigma \dfrac{p_k}{N_k}$.

The equivalent mean constant stress, S_C, for each distribution can be determined by ascertaining from the S-N diagram (Fig. 8) the stress levels associated with the mean life values $\bar{N}$ for the various distributions.

The mean stress, $\bar{S}$, of each distribution is obtained as the sum $\Sigma(p_k \cdot S_{k-1,\,k})$.

In Table V the above characteristics are presented for the four distributions A, B, C, and D. Comparing the trend of the "mean cycle ratios" of Table V with the diagrams in Fig. 9 and with the equivalent mean constant stress values, S_C, representing the "mean life" on these diagrams, as well as with the "mean stress" values $\bar{S}$, the lack of correlation is rather obvious. Thus, for instance, a constant-stress-level fatigue test at $S_C = 46{,}000$ psi would produce a survivorship function roughly equivalent to curve B in Fig. 9. This value of S_C is, however, unrelated to $\bar{S}$ of the load distribution B. Since the effects of overstressing and understressing are eliminated or compensated by the application of a random load sequence with a random starting load in each test, it may be concluded that the linear law of cumulative damage as expressed by $N\ \Sigma\left(\dfrac{p_k}{N_k}\right) =$ constant does not represent an adequate general superposition law of fatigue damage under varying stress levels, no matter what value of the constant is selected; the constant itself appears to be a function of the load spectrum applied. Moreover, the weight in the sum of the individual terms $\left(\dfrac{N p_k}{N_k}\right)$ should not be equal, but depend on the stress level; the linear law apparently underestimates the damage produced at the lower stress levels.

TABLE V.—CHARACTERISTICS OF TEST RESULTS.

	Distribution A	Distribution B	Distribution C	Distribution D
$\Sigma(p_k/N_k)$	1.68×10^{-6}	2.93×10^{-6}	3.93×10^{-6}	5.82×10^{-6}
$N \cdot \Sigma(p_k/N_k)$	1.25	0.51	0.37	0.54
S_C, psi	38 000	46 000	50 000	50 300
S, psi	35 500	37 900	39 400	42 400

Considering the coincidence of the survivorship functions C and D as an indication of this effect, a quick comparison of the cycle ratios at the high and the low stress levels suggests that under the particular test conditions the cycle ratios at the low stress levels would have to be introduced with a weight w_k of roughly 5 to 15 times the weight w_k of the cycle ratios at the high stress levels, if the identity of the sum $\bar{N}\ \Sigma\ \left(\dfrac{w_k p_k}{N_k}\right)$ were to be regarded as an indication of the similarity of total damage.

CONCLUSIONS

The comparatively few preliminary results of random fatigue tests illustrate the use of this type of test as a means of studying the accumulation of fatigue damage under the variable load levels encountered in service. The currently used simple "sum of cycle ratios" does not provide a measure of cumulative damage. It is only through systematically planned random fatigue tests based on general load-distribution functions derived from observations of the important types of loads, that a rational concept of cumulative damage in fatigue may finally emerge.

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DISCUSSION

Mr. W. H. Hoppmann, II.[1]—If I understood the author correctly he said only the Air Force was measuring in service these strains and stresses. I should like to say that in the U.S. Navy about twenty years ago tests were made on the U.S.S. Salinas in the Pacific with the intention of getting a time-strain record for the ships in order to proceed realistically toward design procedure. Further, I should like to mention that Mr. Raymond T. McGoldrick of the David Taylor Basin during World War II devised equipment for counting the strains in certain ranges, that is, bracketed in certain ranges, so that they could relate to design procedure what actually happens on the vessel in operation.

I say this to emphasize the importance of the point Mr. Freudenthal is making, namely, that one should try to relate realistically the actual operating conditions with the fatigue characteristics and requirements.

Mr. A. M. Freudenthal (*author*).—In reply to Mr. Hoppmann I should like to say that relatively extensive and statistically interpretable service-load records have, to my knowledge, only been published with respect to aircraft. It is interesting to learn that the U.S. Navy has also collected such data, and it would be very desirable that a statistical analysis of these data should be made, to establish the character of the distributions. I believe that a serious effort to put fatigue testing on a rational basis, by making the testing conditions representative of the expected loading conditions, is urgently required if the very large and growing volume of fatigue data is ever to be converted into useful engineering information. This can, however, not be done unless the types of random variation of the service conditions, characteristic of various types of engineering structures, are sufficiently well known to permit the establishment of representative random time-series of fatigue loadings.

Therefore, in the interest of establishing more effective and useful fatigue testing procedures, considerable weight should be placed on the collection and rational evaluation of service load records for all types of structures.

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THE CORROSION OF BERYLLIUM COPPER STRIP IN SEA WATER AND MARINE ATMOSPHERES*

By John T. Richards[1]

Synopsis

Results are reported for corrosion tests on beryllium copper and beryllium-cobalt-copper conducted over the past two years at Harbor Island and Kure Beach, N. C. As a means of evaluation, strip specimens were exposed to quiet and flowing sea water, rotation through the water at high velocity, jet impingement, and galvanic action. Specimens were also submitted to marine atmosphere at 80 and 800 ft from the sea.

In addition, the effects of time, velocity, temper, heat treatment, and edge condition were investigated. Comparison materials included phosphorus-deoxidized copper, arsenical admiralty, iron-modified 70-30 cupro-nickel, and phosphor bronze (Grade A). Results are expressed in terms of weight loss and decrease in strength and elongation.

The selection of beryllium copper by designers is frequently dependent upon its ability to resist corrosion. Although some information on the corrosion resistance of beryllium copper has been accumulated over the past 25 years (**1**),[2] available results are based upon different test conditions with alloys of widely varying compositions.

Accordingly, an extensive test program has been initiated to provide a better evaluation of the corrosion resistance of commercial beryllium copper alloys. It is the purpose of this paper to cover corrosion by sea water and marine atmospheres.

Results are presented for complete and partial immersion in sea water, velocity effects as measured by the rotating-spindle and Engineering Experiment Station (EES) methods, jet impingement, galvanic corrosion, and exposure to marine atmospheres. Through the courtesy of The International Nickel Co., Inc., sea water tests were conducted at Harbor Island, N. C., and atmospheric studies at Kure Beach, N. C.

Test Materials

Beryllium copper and beryllium-cobalt-copper strip were tested in various heat-treatable and heat-treated conditions. Comparison materials, including phosphorus-deoxidized copper, phosphor bronze (Grade A), arsenical admiralty, and iron-modified 70-30 cupro-nickel, were furnished by The American Brass Co. Table I lists the composition, condition, and engineering properties of test materials.

Although strip 0.050 in. thick was employed for most tests, other sizes were

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Chief Engineer, Penn Precision Products, Inc., Reading, Pa.; formerly, with The Beryllium Corp., Reading, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 927.

TABLE I.—TEST MATERIALS.

	Beryllium Copper				Beryllium-Cobalt-Copper		Phosphorus-Deoxidized Copper	Phosphor Bronze (Grade A)	Arsenical Admiralty	70-30 Cupro-Nickel (High Iron)
Composition, per cent										
Beryllium	1.85 to 1.95				0.46 to 0.63		...	...	...	...
Cobalt	0.24 to 0.29				2.66 to 2.73		...	...	...	...
Iron	0.05 to 0.13				0.07 to 0.08		...	0.00 to 0.01	0.02	0.40 to 0.48
Silicon	0.09 to 0.11				0.05 to 0.08		...	...	...	...
Aluminum	0.04 to 0.05				0.02 to 0.05		...	...	...	...
Nickel	0.01 to 0.03				0.02 to 0.04		...	...	...	29.78 to 30.42[c]
Tin	0.01 to 0.02				0.01		...	4.67 to 5.42	1.05	...
Lead	0.000				0.000		...	0.01	0.02	0.01
Zinc	0.000				0.00		...	0.00	27.59[c]	0.0
Arsenic	...				...		...	...	0.05	...
Manganese	...				...		...	...	...	0.51 to 0.58
Phosphorus	...				...		0.017 to 0.035	0.015 to 0.017	...	...
Silver	...				0.00 to 0.30		b	...	...	...
Copper	97.42 to 97.71[c]				96.08 to 96.45[c]		99.956 to 99.962[b]	94.39 to 95.17	71.27	68.61 to 69.30
Condition[a]	A	H	AT	HT	H	HT	A	A	A	A
Specific gravity, g per cu cm	8.286	8.284	8.324	8.317	8.778	8.786	8.941[d]	8.858[d]	8.515[d]	8.941[d]
Density, lb per cu in.	0.299	0.299	0.301	0.301	0.317	0.317	0.323[d]	0.320[d]	0.308[d]	0.323[d]
Electrical conductivity, per cent. IACS	16.4	16.9	22.6	23.3	20.0	46.8	91.3	15.1	24.4	4.6
Tensile properties										
Ultimate strength, psi	68 400	113 600	178 800	196 000	66 400	134 500	31 700	47 600	58 500	60 200
Yield strength (0.2 per cent offset), psi	36 300	108 600	147 800	178 400	64 200	121 700	14 000	19 800	28 500	25 900
Proportional limit (0.002 per cent offset), psi	24 100	57 400	109 600	128 900	31 400	84 700	6 600	10 200	18 700	10 700
Elongation in 2 in., per cent	48	5	5	3	5	11	42	55	45	31
Young's modulus, 1-000-000 psi	17.24	16.71	18.79	18.17	18.10	19.82	16.99	15.33	16.35	19.31
Rockwell hardness	B63	B100	C39	C41	B79	C29	F44	B25	B48	B50

[a] A = annealed or solution treated (beryllium copper).
H = cold-rolled 37 per cent reduction in thickness.
AT = solution treated and aged 3 hr at 600 F.
HT = cold-rolled and aged 2 hr at 600 F.

[b] Copper plus silver.

[c] By difference.

[d] Catalog value.

required for several phases of the investigation. Where necessary to use more than one lot of material due to thickness or retest requirements, a composition range is shown in the table. Property data represent average values for the several lots.

TEST METHODS AND RESULTS

The test period extended from October, 1950, to October, 1952. Although weather records for this period and location are not available, an indication can be obtained from the 1952 summary for nearby Wilmington, N. C., which follows:

High	104 F
Low	20 F
Average daily maximum	74.2 F
Average daily minimum	54.3 F
Precipitation	48.11 in.

The water temperature ranged from 51 to 86 F.

All specimens were cleaned, analyzed, measured, and weighed prior to exposure. Some care was exercised in preventing the formation of beryllium oxide. Films of this oxide, which substantially reduce corrosion rates, would produce misleading results (1).

Following exposure, specimens were either hand or chemically cleaned before reweighing. Hand cleaning involved scrubbing with a mild abrasive, whereas chemically cleaned specimens were cathodically pickled in warm, dilute sulfuric acid and scrubbed. On the basis of control specimens, it is unlikely that cleaning losses would have a significant effect upon calculated corrosion rates.

Determinations of tensile strength and percentage elongation in 2 in. were in accordance with the Tentative Methods of Tension Testing of Metallic Materials (E 8 - 52 T),[3] using cross-milled specimens similar to Fig. 5 of that method. The tensile strength was determined from the breaking load, employing the actual thickness and width of the test section.

In sea water immersion and atmospheric tests, sets of four specimens were used. Although maximum and minimum weight losses are given in the tables, the average weight loss for all four specimens has been employed in calculating corrosion rates. Similarly, maximum and minimum tensile values have been reported, but the percentage change is based upon the averages for exposed and storage specimens. These latter specimens were stored at ordinary room temperatures but sealed to prevent atmospheric corrosion. Sets of four storage specimens were tested at the beginning of the program and each time that exposure specimens were removed from the test racks.

Sea Water at Low Velocity:

To investigate the behavior of beryllium copper in low-velocity sea water under fouling conditions, tension specimens 0.050 by 0.5 by 12 in. were immersed in slowly moving sea water (tidal flow). Specimens were mounted vertically on small racks using monel nuts and bolts and were insulated from the rack and fasteners by bakelite insulators. In addition to solution-treated and age-hardened (AT) beryllium copper, comparison specimens of phosphorus-deoxidized copper, arsenical admiralty, and iron-modified 70-30 cupro-nickel were exposed in the annealed (A) condition.

On October 30, 1950, the racks were immersed from the dock at Harbor Island, N. C., to a depth ranging from 2 ft at low tide to 7 ft at high tide. When racks were removed after either 197 or 585 days, specimens were rated for

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[3] 1952 Book of ASTM Standards, Part 2, p. 1208.

TABLE II.—WEIGHT LOSS RESULTING FROM IMMERSION IN SEA WATER AT LOW VELOCITY.
Harbor Island, N. C., October, 1950, to June, 1952.
Tension specimen, exposed area 16.6 sq. in. or 1.07 sq. dm.

Material and Condition	Exposure Time, days	Weight Loss, g		Corrosion Rate		Pitting, in.	Remarks
		Min	Max	mdd*	in. per yr		
Beryllium copper (AT)	92	0.73	0.80	8	0.0013	0 to 0.005	...
	197	1.49	2.24	9	0.0015	0 to 0.012	No fouling
	584	2.72	3.21	5	0.0008	0 to 0.012	Slight surface corrosion, edges good
Phosphorus-deoxidized copper (A)	197	1.68	1.91	9	0.0015	0 to 0.001	No fouling
	585	3.16	3.98	5	0.0008	...	Slight surface corrosion, edges bad
Arsenical admiralty (A)	197	1.77	1.99	9	0.0015	None	No fouling
	584	2.93	3.56[a]	5	0.0008	...	Slight surface corrosion, edges corroded
Cupro-nickel, iron-modified 70-30 (A)	197	0.25	0.29	1	0.0002	None	Fouling
	584	0.50	0.73	1	0.0002	0 to 0.0085 under tiny barnacles	Good condition, slight etch or pits under tiny barnacles

[a] One specimen corroded in half and is not included in results.
* Milligrams per square decimeter per day.

TABLE III.—CHANGE IN TENSILE PROPERTIES RESULTING FROM IMMERSION IN SEA WATER AT LOW VELOCITY.
Harbor Island, N. C., October, 1950, to June, 1952.
Exposed as tension specimens.

Material and Condition	Exposure Time, days	Storage Specimens				Exposed Specimens				Change, per cent	
		Tensile Strength, psi,		Elongation in 2 in., per cent		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength	Elongation
		Min	Max	Min	Max	Min	Max	Min	Max		
Beryllium copper (AT)	0	164 700	170 000	5	5						
	92	180 000[a]	183 500[a]	4[a]	5[a]	179 200[a]	181 000[a]	5[a]	6[a]	−0.8	0
	197	163 300	170 800	4	6	153 800	160 500	5	6	−6.0	+20
	584	171 000	176 200	3	5	165 200	170 000	2	5	−3.1	−25
Phosphorus-deoxidized copper (A)	0	30 700	31 500	36	40						
	197	31 100	31 900	41	43	30 800	31 300	39	41	−1.9	−5
	584	32 100	32 800	36	40	31 300	31 900	27	37	−2.8	−18
Arsenical admiralty (A)	0	54 300	57 200	43	45						
	197	57 800	58 700	45	45	56 700	57 400	42	44	−1.5	−4
	584	58 200	58 700	41	42	56 700[b]	58 400	36[b]	40	−1.9	−10
Cupro-nickel, iron-modified 70-30 (A)	0	59 700	60 000	28	30						
	197	58 400	59 300	29	31	58 800	59 300	29	30	0	−6
	584	60 200	61 200	28	28	59 400	60 000	24	28	−1.8	−7

[a] Different lot having an original tensile strength of 188, 100 psi with 5 per cent elongation.
[b] One specimen corroded in half and is not included in results.

fouling, cleaned, weighed, inspected for pitting, and finally tension tested. The results are given in Tables II and III.

These data indicate that beryllium copper, copper, and admiralty behave similarly and are corroded at a rate greater than cupro-nickel. The higher corrosion rate of the former materials prevents fouling; however, fouling was observed in the case of cupro-nickel. Both beryllium copper and copper showed a tendency toward crevice corrosion, while the more severe edge corrosion noted for copper and admiralty may be responsible for the lower elongation values of these materials.

It should be pointed out that the beryllium copper specimens were exposed in the precipitation-hardened (AT) condition whereas the other materials were annealed (A), so that the strength of the beryllium copper ranged from approximately three to six times that of the other materials. In order to determine whether the high strength of the precipitation-hardened condition might affect corrosion rates or lead to reduced tensile values as a result of notching, an additional series of beryllium copper specimens (A, H, AT, HT conditions) was exposed for 92 days. In addition to tension specimens, rectangular strips were also included. Since the rectangular strips were machined into tension specimens after exposure, the influence of edge corrosion could be evaluated.

TABLE IV.—EFFECT OF CONDITION OF BERYLLIUM COPPER ON WEIGHT LOSS RESULTING FROM IMMERSION IN SEA WATER AT LOW VELOCITY.

Harbor Island, N. C., June, 1952, to September, 1952 (92 days).
Tension specimen, exposed area 16.5 sq in. or 1.07 sq dm.
Rectangular specimen, exposed area 16.8 sq in. or 1.08 sq dm.

Condition	Specimen Shape	Weight Loss, g		Corrosion Rate		Pitting, in.
		Min	Max	mdd	in. per yr	
A	Tensile	0.76	0.83	8	0.0013	0 to 0.0005
	Rectangular	0.87	0.93	9	0.0015	0 to 0.002
H	Tensile	0.74	0.84	8	0.0013	0 to 0.003
	Rectangular	0.81	0.94	9	0.0015	0 to 0.002
AT	Tensile	0.73	0.80	8	0.0013	0.003 to 0.011
	Rectangular	0.73	0.82	8	0.0013	0.001 to 0.011
HT	Tensile	0.72	0.91	8	0.0013	0.004 to 0.011
	Rectangular	0.75	0.88	8	0.0013	0 to 0.007

TABLE V.—EFFECT OF CONDITION OF BERYLLIUM COPPER ON CHANGE IN TENSILE PROPERTIES RESULTING FROM IMMERSION IN SEA WATER AT LOW VELOCITY.

Harbor Island, N. C., June, 1952, to September, 1952 (92 days).
Tension specimen, tested as exposed.
Rectangular specimen, milled to tension specimen after exposure.

Condition	Exposed Shape	Storage 92 days[a]				Exposed 92 days				Change, per cent	
		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength	Elongation
		Min	Max	Min	Max	Min	Max	Min	Max		
A	Tensile	69 400	71 000	45	48	67 300	71 800	42	46	−1.6	−2
	Rectangular	69 400	71 000	45	48	68 200	68 500	45	46	−2.8	0
H	Tensile	113 500	115 200	4	5	108 000	110 000	4	5	−4.8	0
	Rectangular	113 500	115 200	4	5	112 300	118 000	6	8	−0.3	+40
AT	Tensile	180 000	183 500	4	5	179 200	181 000	5	6	−0.2	0
	Rectangular	180 000	183 500	4	5	180 500	182 300	5	7	+0.6	+20
HT	Tensile	193 000	195 800	3	4	187 500	193 500	2	3	−1.9	−25
	Rectangular	193 000	195 800	3	4	194 000	196 500	4	5	+0.2	+25

[a] Original properties:

Condition	Tensile strength, psi		Elongation in 2 in., per cent	
	Min	Max	Min	Max
A	68 400	70 900	42	50
H	112 800	115 000	5	5
AT	187 000	189 000	5	6
HT	196 100	197 900	2	3

As indicated in Tables IV and V, material condition had no marked effect upon corrosion rates as measured by weight loss or change in tensile properties. In fact, weight losses for precipitation-hardened specimens were slightly less than for heat-treatable stock, although pitting was more severe. As anticipated, specimens with corroded edges showed a slightly greater loss in tensile strength and elongation.

Table VI affords a comparison between the results of the present project and similar but earlier work with low-velocity sea water by other investigators. The order of agreement appears fair.

TABLE VI.—COMPARISON OF SEVERAL INVESTIGATIONS OF CORROSION RATES IN SEA WATER AT LOW VELOCITY (STRIP OR SHEET SPECIMENS).

Material	Investigation	Location	Nominal Composition, per cent[a]	Condition	Exposure Time, days	Corrosion Rate		Change, per cent	
						mdd	in. per yr	Tensile Strength	Elongation
Beryllium copper......	Present	Harbor Island, N. C.	1.90 Be, 0.26 Co	AT	584	5	0.0008	−3.1	−25
	Tracy (2)	Long Island Sound, Conn.	2.00 Be, 0.35 Ni	AT	300[b]	2	0.0004	...	...
Phosphorus-deoxidized copper...............	Present	Harbor Island, N. C.	0.025 P	A	584	5	0.0008	−2.8	−18
	Bulow (3)	Kure Beach, N. C.	0.009 P	A	388	9	0.0015	...	...
	Tracy, Thompson, and Freeman (4)	Long Island Sound, Conn.	0.020 P	A	730[b]	4	0.0007	−7.0	−11
	ASTM (5)	Bridgeport, Conn.	0.007 P	A	243	15	0.0025	−14.4	−26
Arsenical admiralty....	Present	Harbor Island, N. C.	27.6 Zn, 1.05 Sn, 0.05 As	A	584	5	0.0008	−1.9	−10
	Bulow (2)	Kure Beach, N. C.	27.6 Zn, 1.12 Sn, 0.04 As	A	388	9	0.0015	...	...
Cupro-nickel, iron-modified 70-30........	Present	Harbor Island, N. C.	30.0 Ni, 0.44 Fe	A	584	1	0.0002	−1.8	−7
	Bulow (2)	Kure Beach, N. C.	28.9 Ni, 0.42 Fe	A	388	2	0.0003	...	...

[a] Balance copper.

[b] Specimens mounted at one-half tide (alternately immersed twice daily).

Flowing Sea Water:

To obtain the desired test conditions at moderate velocities, sea water is pumped through a trough at 2 to 4 ft per sec. Insulated tension specimens were suspended in the trough approximately 45 deg from a vertical position. Certain specimen sets were partially immersed to their midpoint to determine the "water-line effect," while others were totally submerged.

Test results are presented in Tables VII, VIII, IX, and X, while typical specimens that were totally immersed in flowing sea water for 21 months are shown in Fig. 1. Based upon loss of weight, width, tensile strength, and elongation, optimum resistance to corrosion in flowing sea water is obtained with cupro-nickel, followed by beryllium copper, copper, and admiralty in the order named.

Pitting and impingement corrosion of the leading edge seem to be limited to admiralty and copper. These types of attack are probably responsible for the marked reduction in tensile strength and elongation noted in the case of copper and admiralty. Table IX indicates that copper offers better resistance to edge corrosion than admiralty under the particular conditions of this test. This conclusion, however, does not necessarily agree with service experience, since admiralty has proven satisfactory as a condenser tube material handling sea water, whereas copper has not.

TABLE VII.—WEIGHT LOSS RESULTING FROM PARTIAL IMMERSION IN FLOWING SEA WATER AT MODERATE VELOCITY.

Harbor Island, N. C., October, 1950, to September, 1952.

Tension specimen, exposed area 0.64 sq dm.

Material and Condition	Exposure Time, days	Weight Loss, g		Corrosion Rate		Loss of Width, in.[a]	Loss of Thickness, in.[b]	Remarks
		Min	Max	mdd	in. per yr			
Beryllium copper (AT)	92	1.37	1.44	24	0.0040	...	...	Leading edge grooved
	274	2.27	2.77	14	0.0023	...	...	Slight corrosion
	697	2.94	3.42	7	0.0011	0.000	0.004	Edges and surface slightly corroded
Phosphorus-deoxidized copper (A)	92	1.00	1.38	20	0.0030	...	...	...
	274	3.53	4.50	22	0.0035	...	...	Pitting up to 0.011 in.
	697	4.17	5.84	11	0.0018	0.125	0.007	Edges and surface badly corroded
Arsenical admiralty (A)	92	1.14	1.41	21	0.0030	...	...	...
	274	3.70	4.88	23	0.0038	...	...	Pitting up to 0.013 in.
	697	5.20	7.80	15	0.0025	0.125	0.008	Edges and surface badly corroded
Cupro-nickel, iron-modified 70-30 (A)	92	0.22	0.37	5	0.0008	...	...	...
	274	0.37	0.47	2	0.0004	...	...	Slight corrosion
	697	0.60	0.81	2	0.0003	0.000	0.000	Surface slightly etched, edges good

[a] Due to impingement corrosion of leading edge.

[b] Loss of thickness at bottom or submerged end compared to top of specimen.

TABLE VIII.—CHANGE IN TENSILE PROPERTIES RESULTING FROM PARTIAL IMMERSION IN FLOWING SEA WATER AT MODERATE VELOCITY.

Harbor Island, N. C., October, 1950, to September, 1952.

Exposed as tension specimens.

Material and Condition	Exposure Time, days	Storage Specimens				Exposed Specimens				Change, per cent	
		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength	Elongation
		Min	Max	Min	Max	Min	Max	Min	Max		
Beryllium copper (AT)	0	164 700	170 000	5	5						
	92	166 100	169 100	4	5	165 500	168 500	7	8	−0.5	+100
	274	167 800	170 200	5	5	161 200	166 900	4	6	−2.7	0
	697	170 100	173 000	4	4	162 000	166 100	5	6	−4.2	+50
Phosphorus-deoxidized copper (A)	0	30 700	31 500	36	40						
	92	30 600	31 800	38	43	31 200	32 100	39	44	+1.0	0
	274	31 700	32 500	40	44	27 900	29 800	24	31	−10.9	−37
	697	32 500	32 800	41	43	26 700	29 000	20	22	−13.8	−50
Arsenical admiralty (A)	0	54 300	57 200	43	45						
	92	57 100	57 700	43	45	57 000	58 300	42	45	+0.5	−2
	274	57 900	58 700	45	46	51 000	54 400	28	34	−10.7	−31
	697	58 500	59 000	43	43	39 200	54 600	18	30	−18.7	−44
Cupro-nickel, iron-modified 70-30 (A)	0	59 700	60 000	28	30						
	92	58 300	58 600	29	30	58 700	59 500	27	29	+1.4	−7
	274	59 100	59 500	28	30	58 700	60 200	29	30	−0.2	+3
	697	60 100	60 600	29	30	59 400	60 000	29	30	−1.2	0

Although there is little difference in weight loss, there is slightly greater decrease in tensile properties for specimens partially immersed compared with those totally submerged. This may be due to the more severe edge attack resulting from partial immersion.

The data presented make possible a comparison of the corrosion rates be-

TABLE IX.—WEIGHT LOSS RESULTING FROM TOTAL IMMERSION IN FLOWING SEA WATER AT MODERATE VELOCITY.

Harbor Island, N. C., October, 1950, to September, 1952.

Tension specimen, exposed area 0.44 sq dm.

Material and Condition	Exposure Time, days	Weight Loss, g		Corrosion Rate		Loss of Width, in.[a]	Loss of Thickness, in.[b]	Remarks
		Min	Max	mdd	in. per yr			
Beryllium copper (AT)	92	0.99	1.01	25	0.0040	...	...	...
	274	1.31	1.50	12	0.0019	...	...	Slight corrosion
	697	2.15	2.28	7	0.0011	0.000	0.005	Edges and surface slightly corroded
Phosphorus deoxidized copper (A)	92	0.92	1.01	24	0.0040	...	...	...
	274	1.90	2.42	18	0.0029	...	...	Average pitting 0.002 to 0.005 in.
	697	3.42	4.19	12	0.0020	0.000	0.005	Edges and surface badly corroded
Arsenical admiralty (A)	92	0.91	0.97	23	0.0040	...	...	...
	274	1.87	2.52	19	0.0037	...	...	Average pitting 0.007 to 0.009 in.
	697	4.09	6.52	17	0.0029	0.125	0.008	Edges and surface badly corroded
Cupro-nickel, iron-modified 70-30 (A)	92	0.25	0.32	7	0.0010	...	...	...
	274	0.34	0.36	3	0.0005	...	...	Slight corrosion
	697	0.50	0.61	2	0.0003	0.000	0.000	Slight corrosion under barnacles

[a] Due to impingement corrosion of leading edge.
[b] Loss of thickness at bottom compared to top under spacer.

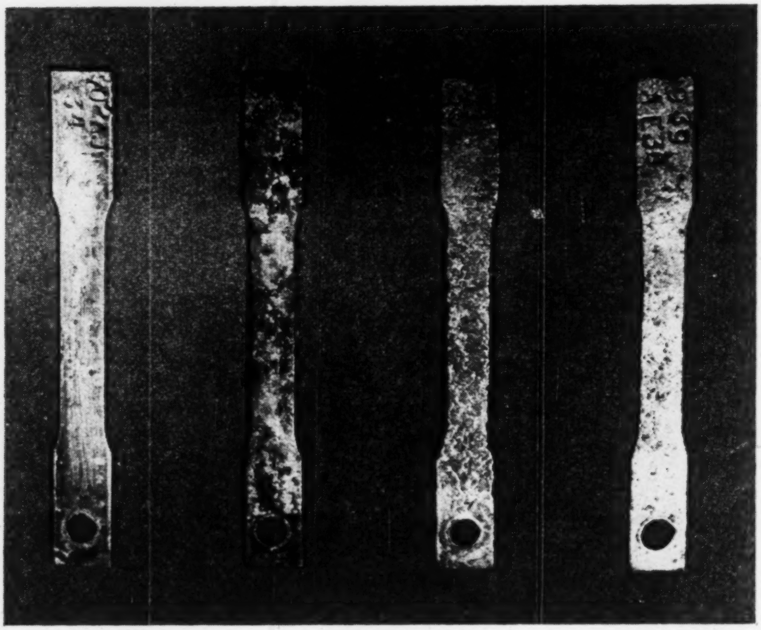


FIG. 1.—Specimen After Total Immersion in Flowing Sea Water (2 to 4 ft per sec) for 21 months. From left: cupro-nickel, beryllium copper, admiralty, and copper.

tween relatively quiet (tidal flow) and flowing sea water (2 to 4 ft per sec). Although velocity exerts a strong influence upon weight loss, as shown in Fig. 2, the effect upon tensile properties is not consistent and is of smaller magnitude. In the case of copper and admiralty, pitting and erosion of the leading edge become important factors with velocity increases.

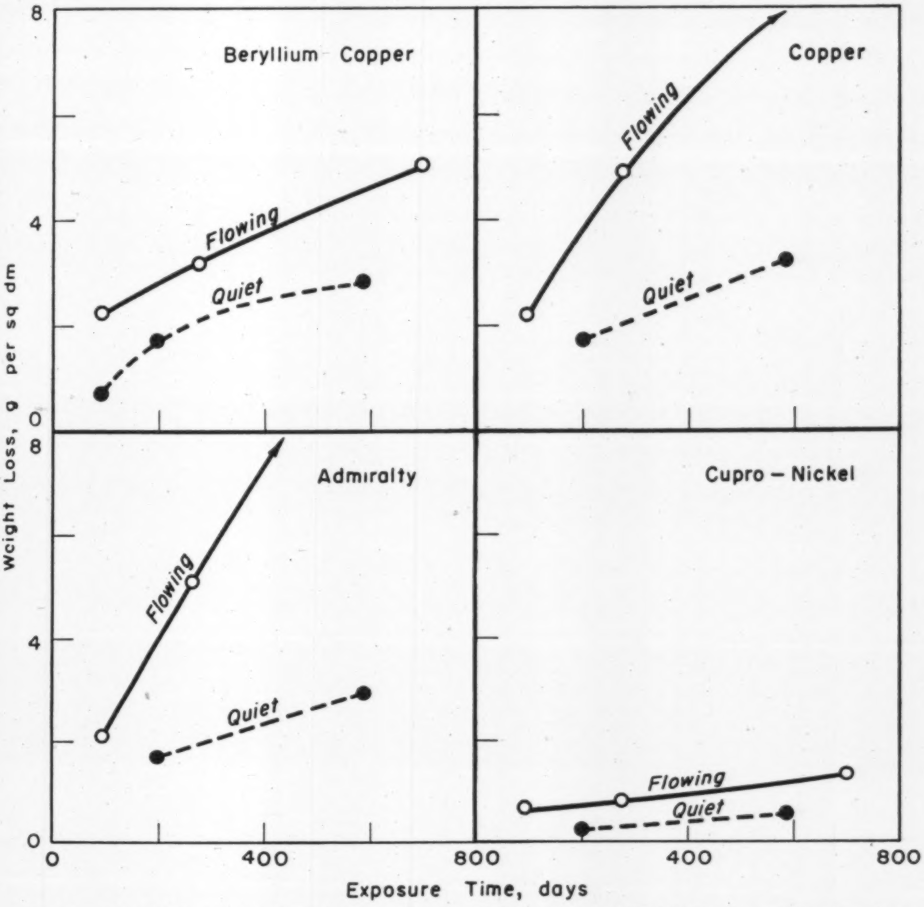


FIG. 2.—Weight Loss as Affected by Sea Water Velocity: Quiet (Tidal Flow) and Flowing (2 to 4 ft per sec).

Rotating-Spindle Apparatus:

To determine the effect of increased velocities upon corrosion rates, disk specimens were tested in the rotating-spindle apparatus (6). Specimens 5 in. in diameter and 0.050 to 0.125 in. thick were held by suitable insulated fasteners 6 in. below the surface of sea water on vertical shafts. A shaft speed was selected to give calculated linear velocities ranging up to 28 ft per sec. After a test period of 60 days, specimens were cleaned, weighed, and the thickness measured at different radii to determine the depth of attack at various velocities.

In the first run, two beryllium copper

TABLE X.—CHANGE IN TENSILE PROPERTIES RESULTING FROM TOTAL IMMERSION IN FLOWING SEA WATER AT MODERATE VELOCITY.

Harbor Island, N.C., October, 1950, to September, 1952.

Exposed as tension specimens.

Material and Condition	Exposure Time, days	Storage Specimens				Exposed Specimens				Change, per cent	
		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength	Elongation
		Min	Max	Min	Max	Min	Max	Min	Max		
Beryllium copper (AT)	0	164 700	170 000	5	5						
	92	166 100	169 100	4	5	168 500	172 500	4	5	+1.8	+25
	274	167 800	170 200	5	5	169 500	174 000	4	6	+1.1	0
	697	170 100	173 000	4	4	162 800	167 300	3	4	−3.7	0
Phosphorus-deoxidized copper (A)	0	30 700	31 500	36	40						
	92	30 600	31 800	38	43	31 500	32 500	40	42	+2.9	0
	274	31 700	32 500	40	44	29 600	31 000	29	41	−6.5	−17
	697	32 500	32 800	41	43	30 000	31 700	28	33	−5.2	−26
Arsenical admiralty (A)	0	54 300	57 200	43	45						
	92	57 100	57 700	43	45	57 700	58 400	43	45	+1.4	−2
	274	57 900	58 700	45	46	53 500	56 000	35	45	−5.5	−11
	697	58 500	59 000	43	43	42 700	50 000	20	30	−21.3	−47
Cupro-nickel, iron-modified 70-30 (A)	0	59 700	60 000	28	30						
	92	58 300	58 600	29	30	58 700	59 500	29	31	+1.2	0
	274	59 100	59 500	28	30	58 700	59 400	28	30	−0.7	0
	697	60 100	60 600	29	30	58 700	59 800	23	30	−1.7	−10

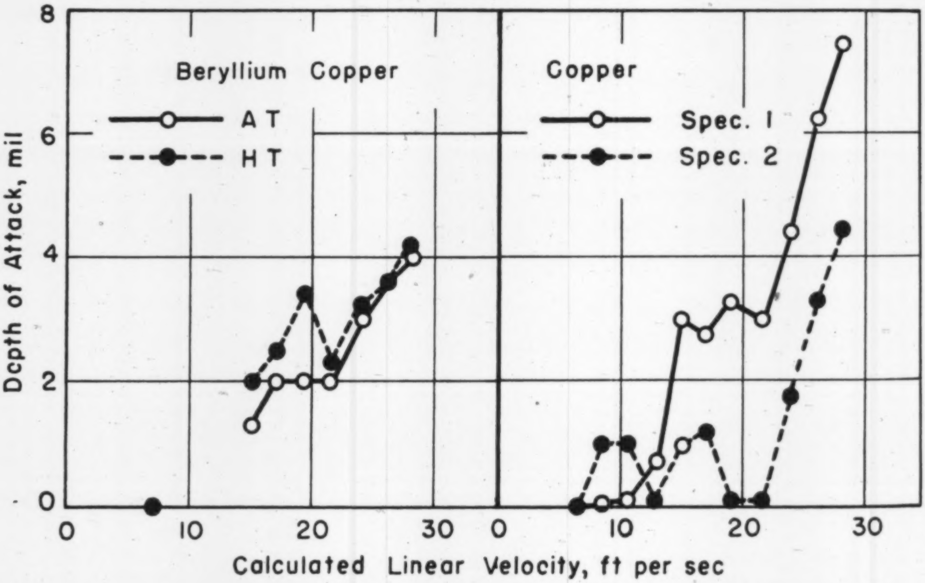
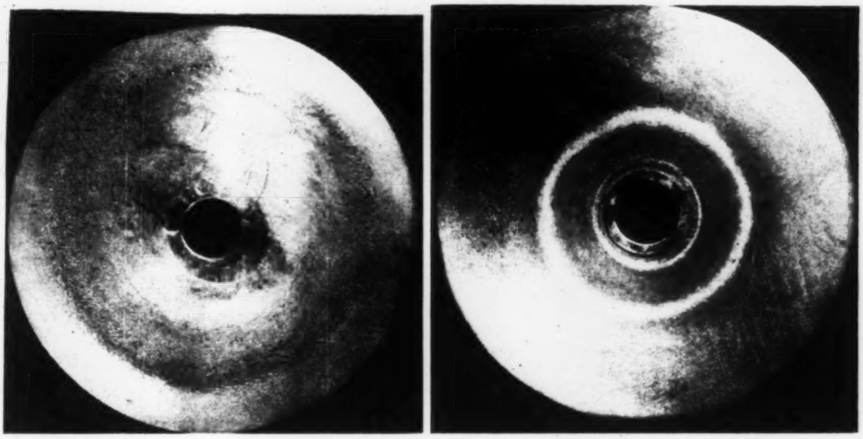


FIG. 3.—Effect of Velocity on Depth of Attack in Rotating-Spindle Test for Beryllium Copper (AT and HT Conditions) and Copper (2 Annealed Specimens).

specimens (AT and HT conditions) were tested in sea water having an average temperature of 54 F. These specimens were resurfaced and subsequently retested with two annealed copper disks in sea water averaging 81 F. It is of interest to note that the over-all corrosion rate of the beryllium copper disks exposed at 54 F was slightly less than 50 per cent of the value obtained at 81 F (59 to 136 mdd (milligrams per sq decimeter per day)).

Figure 3 illustrates the effect of velocity on depth of attack, and typical disks are shown in Fig. 4. The critical velocity or speed at which the protective film loses its effectiveness is approximately 15 ft per sec for both copper and beryllium copper. This figure can be compared with critical velocities ranging from 15.0 to 17.5 ft per sec obtained in earlier tests on admiralty and various cupro-nickel alloys (7). Although these latter alloys maintain their protective films at slightly higher velocities, once the film is lost the depth of the attack at higher speeds (18 to 25 ft per sec) is greater than that observed with either copper or beryllium copper. It should be pointed out, however, that the critical velocity in this test may vary from that encountered with a fixed specimen and moving water, since disk velocities vary from point to point.



(*a*) Copper (A). (*b*) Beryllium copper (AT).

FIG. 4.—Typical Disk Specimens from Rotating-Spindle Test in Sea Water.

Engineering Experiment Station (EES) Apparatus:

As a further means of checking velocity effects and resistance to erosion, flat bar specimens were tested in the EES machine (6, 8). Specimens, which are attached by insulated fasteners to plastic disks mounted on a horizontal shaft, are rotated at high velocity in a tank containing sea water. By virtue of violent agitation produced by the whirling disks and test bars, large amounts of air are entrained in the water. By suitable means of control, the water temperature is held to ±2 C. Exhaustion of corrosive constituents or accumulation of corrosion products is prevented by the continuous passage of fresh sea water through the tank.

TABLE XI.—WEIGHT LOSS RESULTING FROM EXPOSURE IN EES APPARATUS FOR 60 DAYS.

Harbor Island, N. C., February, 1952, to April, 1952.

Nominal tip velocity, 27 ft per sec. Specimen size, approximately 4 by ¾ by ¼ in.

Material	Average Sea Water Temperature, deg Cent	Condition	Corrosion Rate	
			mdd	in. per yr
Beryllium copper......	20	AT	78	0.013
		HT	77	0.013
Arsenical admiralty....	15	A	125	0.021
	26	A	133	0.022
Cupro-nickel, iron-modified 70-30.......	30	A	31	0.005

Test results are listed in Table XI. In this instance, data for the comparison alloys were obtained from another investigation but are included because of the similarity of test conditions (nominal tip velocity, 27 ft per sec, and specimen size, approximately $\frac{1}{4}$ by $\frac{3}{4}$ by 4 in.).

Typical beryllium copper specimens in the AT and HT conditions are shown in Fig. 5. Although erosion is more apparent in the case of the AT bar as evidenced by surface roughening, the corrosion rates given in Table XI are similar. The influence of velocity can be readily appreciated by comparing the values for 92-day immersion in flowing sea water (2 to 4 ft per sec) from Table IX with the EES results (27 ft per sec) in Table XI. In the case of beryllium copper, the velocity increase produces a rise in corrosion rate from 25 to 78 mdd. Similar increases were observed for admiralty (23 to 129 mdd) and cupro-nickel (7 to 31 mdd).

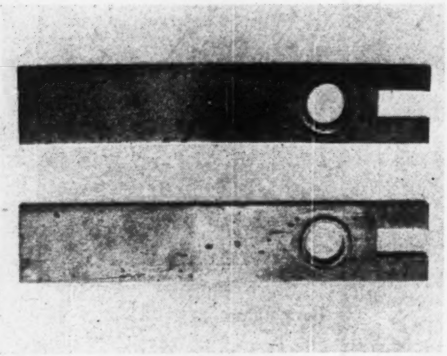


FIG. 5.—Typical Beryllium Copper Bar Specimens After Test in EES Apparatus.
(*Top*) AT condition.
(*Bottom*) HT condition.

Sea-Water Jet Tests:

Two different jet tests, the aspirator and the British types, have been employed to determine the resistance to impingement attack by an aerated stream of sea water. In the aspirator test, air bubbles are sucked into the jet through a venturi-type nozzle and the resulting aerated stream is directed against the test specimen (6). The British-type jet premixes air and sea water in a manifold under controlled conditions. The mixture is then delivered to a submerged specimen holder where it passes through a standard jet to impinge against the specimen (9).

TABLE XII.—RESULTS OF EXPOSURE IN ASPIRATOR-TYPE SEA WATER JET TEST APPARATUS FOR 30 DAYS.

Harbor Island, N. C., October, 1950, to November, 1950.
Average sea water temperature 14.4 C. 2.4 per cent by volume air added.
Peak jet velocity, 12 ft per sec. Specimen size, 1.5 by 1.5 in.

Material	Condition	Weight Loss, mg per day	Maximum Depth of Attack, in.	Diameter of Attacked Area, in.
Beryllium copper	A	12.8	0.0030	0.19
	H	14.3	0.0043	0.19
	AT	11.5	0.0023	0.50
	HT	10.9	0.0043	0.22
Beryllium-cobalt-copper	H	9.7	0.0025	0.19
	HT	14.0	0.0035	0.25
Phosphorus-deoxidized copper	A	9.8	0.0030	...
Arsenical admiralty	A	12.9	0.0035	0.88
Cupro-nickel, iron-modified 70-30	A	5.8	0.0030	0.38

TABLE XIII.—RESULTS OF EXPOSURE IN BRITISH TYPE JET TEST APPARATUS FOR 30 DAYS.

Harbor Island, N. C., November, 1950, to December, 1950.
Average sea water temperature, 9.3 C. 2 to 4 per cent by volume air added.
Peak jet velocity, 15 ft per sec. Specimen size, 3⅜ by ¾ in.

Material	Condition	Weight Loss, mg per day	Maximum Depth of Attack, in.	Diameter of Attacked Area, in.
Beryllium copper	A	10.8	0.0046	0.67
	H	10.0	0.0020	0.44
	AT	11.4	0.0040	0.67
Beryllium-cobalt-copper	HT	12.8	0.0049	0.50
Phosphorus-deoxidized copper	A	9.9	0.0030	0.58
Phosphor bronze, Grade A	A	7.7	0.0020	0.73

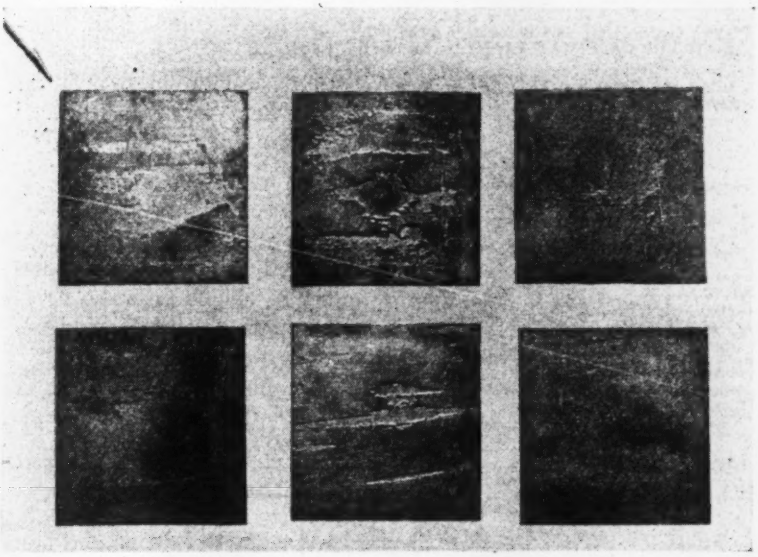


FIG. 6.—Typical Beryllium Copper Specimens After Exposure to Aspirator-Type Sea Water Jet. From left, top: beryllium-cobalt-copper (H) and (HT), beryllium copper (A); bottom: beryllium copper (AT), (H), and (HT).

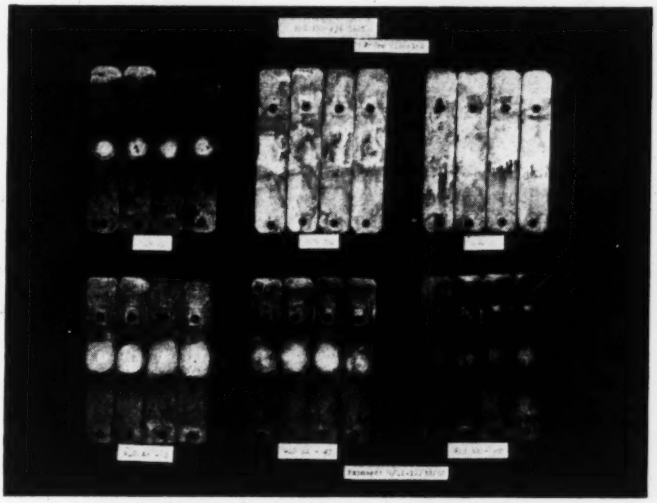


FIG. 7.—Typical Specimens After Exposure to British-Type Sea Water Jet. From left, top: beryllium-cobalt-copper (HT), phosphor bronze (Grade A) (A), and copper (A); bottom: beryllium copper (A), (AT), and (H).

These jet tests were originally devised as a means of evaluating condenser-tube materials, but they are also generally useful in measuring velocity effects and resistance to erosion. This type of attack may prove especially severe, since the continuous removal of the protective film by impingement is greatly abetted by the breaking of the air bubbles entrained in the water at the metal surface.

TABLE XIV.—RELATIONSHIP BETWEEN OPEN CIRCUIT POTENTIALS AND CORROSION RATES OF SEVERAL COPPER-BASE MATERIALS IN SEA WATER.

Velocity, 7.7 to 13.0 ft per sec. Temperature, 20 to 25 C. Exposed area, 2.875 sq in.

	Steady Potential Relative to Saturated Calomel Half Cell, v	Corrosion Rates Calculated from Weight Losses, mdd
Copper	0.36	106
Beryllium copper	0.29	60
Aluminum brass	0.27	25
Admiralty, phosphorus-inhibited	0.27	25
90-10 cupro-nickel, 1.5 Fe	0.27	25
90-10 cupro-nickel, low iron	0.24	22
70-30 cupro-nickel, low iron	0.22	37

Aspirator-type jet test results are reported in Table XII, while typical specimens are shown following cleaning in Fig. 6. Although the depth of attack is approximately the same for all materials, cupro-nickel was found to provide the best resistance in terms of weight loss. This latter result is in agreement with field experience, wherein cupro-nickel has been found to provide better resistance to impingement pitting than either copper or admiralty. Based upon data for materials common to both jet tests, the corrosion rates obtained in the British apparatus (Table XIII and Fig. 7) were similar to those found with the aspirator type.

Galvanic Corrosion:

The galvanic corrosion and potential of beryllium copper strip were determined in flowing sea water. Details of the test apparatus and procedure have been described elsewhere (**7, 10**).

Data obtained in this test are helpful in predicting the direction of galvanic currents when two dissimilar materials are in contact in a suitable electrolyte. Open circuit measurements of the steady potential, negative to a saturated calomel half cell, after a 14-day exposure period in turbulent sea water (7.8 ft per sec and 20 C) give a value of 0.29 v for beryllium copper (AT). This value can be compared with a potential of 0.20 v resulting from immersion in artificial sea water at 25 C (**11**).

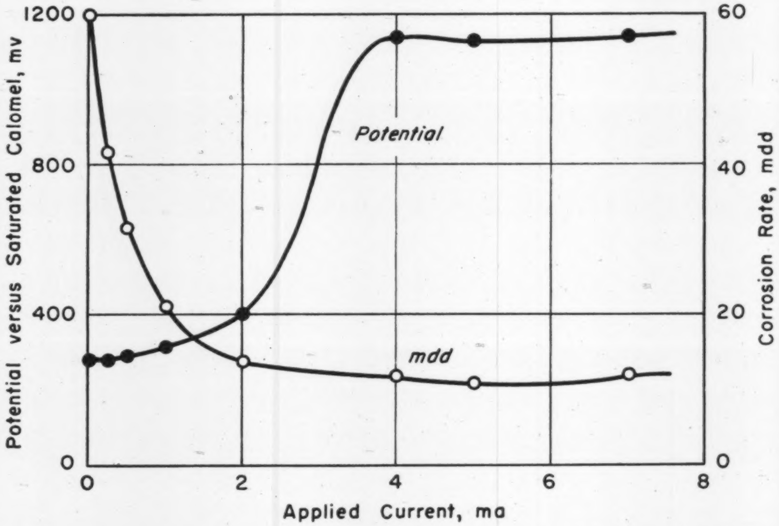


FIG. 8.—Cathodic Polarization of Beryllium Copper (AT) in Sea Water Flowing at 7.8 ft per sec and 20 C.

The relationships between open circuit

potentials and corrosion rates for several copper-base materials are listed in Table XIV. Although data for beryllium copper are based upon a sea water velocity of 7.8 ft per sec and a temperature of 20 C, comparison materials (7) were run at 13 ft per sec and 25 C.

TABLE XV.—WEIGHT LOSS AND CHANGE IN TENSILE PROPERTIES RESULTING FROM EXPOSURE IN MARINE ATMOSPHERES.

Kure Beach, N. C., October, 1950, to October, 1952.
Specimen size: 0.050 by 3 by 12.75 in. Exposure Area: 77.1 sq in. or 5 sq dm.
Exposed 30 deg from horizontal.

Material and Condition	Exposure Time, days	Corrosion Rate			Storage Specimens				Exposed Specimens				Change, per cent	
		Weight Loss, g		mdd	Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength, psi		Elongation in 2 in., per cent		Tensile Strength	Elongation
		Min	Max		Min	Max	Min	Max	Min	Max	Min	Max		
80 FT FROM OCEAN														
Beryllium copper (AT)	0	...	...	...	164 700	170 000	5	5	...	...	...	...	...	...
	361	0.81	1.13	0.5	169 200	171 800	5	6	174 500	180 200	4	6	+4.0	0
	731	1.24	1.29	0.3	170 100	173 000	4	4	176 000	179 000	6	6	+3.8	+50
Phosphorus-deoxidized copper (A)	0	...	...	...	30 700	31 500	36	40	...	...	...	...	...	...
	361	1.29	1.96	0.8	31 600	31 800	41	42	31 700	32 200	40	42	+1.3	−2
	731	1.74	2.04	0.5	32 500	32 800	41	43	32 700	33 800	39	40	+1.8	−5
Phosphor bronze (Grade A) (A)	0	...	...	...	46 000	46 200	54	56	...	...	...	...	...	...
	361	2.18	2.49	1.3	47 500	47 700	52	57	50 700	51 300	53	58	+6.9	0
	731	3.24	3.54	0.9	47 500	47 900	52	54	48 200	50 200	50	55	+2.5	+2
800 FT FROM OCEAN														
Beryllium copper (A)	0	...	...	...	66 000	67 000	46	51	...	...	...	...	...	...
	361	0.41	0.43	0.2	67 100	67 500	48	50	68 800	70 800	36	47	+4.2	−12
	731	0.73	0.74	0.2	68 400	68 700	42	45	68 300	69 000	46	50	+0.1	+9
Beryllium copper (H)	0	...	...	...	111 300	114 200	4	6	...	...	...	...	...	...
	361	0.49	0.55	0.2	112 000	114 100	5	5	113 800	115 000	5	6	+1.2	+20
	731	0.66	0.76	0.2	110 800	116 000	4	5	116 300	117 000	5	7	+2.5	+20
Beryllium copper (AT)	0	...	...	...	164 700	170 000	5	5	...	...	...	...	...	...
	361	0.42	0.45	0.3	169 200	171 800	5	6	178 000	179 000	3	6	+4.5	−33
	731	0.69	0.73	0.2	170 100	173 000	4	4	179 000	180 000	6	6	+4.7	+50
Beryllium copper (HT)	0	...	...	...	190 600	196 200	1	3	...	...	...	...	...	...
	361	0.36	0.40	0.2	193 800	197 000	3	3	196 800	205 500	2	3	+2.1	−33
	731	0.59	0.70	0.2	196 000	196 200	3	3	187 500	198 500	3	4	−0.4	+33
Beryllium-cobalt-copper (HT)	0	...	...	...	127 000	136 000	8	10	...	...	...	...	...	...
	361	0.62	0.66	0.4	134 300	134 700	10	11	129 900	133 200	9	12	−2.4	0
	731	1.05	1.16	0.3	136 700	137 200	9	10	133 500	134 000	10	11	−2.5	+10
Phosphorus-deoxidized copper (A)	0	...	...	...	30 700	31 500	36	40	...	...	...	...	...	...
	361	0.94	1.03	0.6	31 600	31 800	41	42	31 800	32 300	40	42	+1.3	0
	731	1.55	1.61	0.4	32 500	32 800	41	43	33 200	33 400	39	40	+2.1	−7
Phosphor bronze (Grade A) (A)	0	...	...	...	46 000	46 200	54	56	...	...	...	...	...	...
	361	1.19	1.23	0.7	47 500	47 700	52	57	50 600	52 400	49	57	+8.8	−4
	731	1.86	1.92	0.5	47 500	47 900	52	54	48 300	48 500	54	55	+1.5	+2

As indicated by LaQue (7), open circuit potential measurements may prove unreliable in predicting corrosion rates without some knowledge of the polarization characteristics of the materials involved. Consequently, polarization studies were conducted in flowing sea water with controlled current densities applied to the specimens. Figure 8 shows the effect of applied current upon the potential and corrosion rates. A different specimen was employed for each current density.

The accompanying results emphasize the fact that the galvanic behavior of beryllium copper is similar to that observed in other copper materials.

Marine Atmospheres:

To determine resistance to marine atmospheres, specimens were exposed for 2 yr at 80 and 800 ft from the ocean at Kure Beach, N. C. Rectangular specimens, measuring 0.050 by 3.00 by 12.75 in., were inclined 30 deg from the horizontal. Corrosion rates were obtained from the weight differences noted between the original weighing before testing and the final weighing after exposure and cleaning. Tension specimens were subsequently machined from the rectangular strips. In addition to beryllium copper and beryllium-cobalt-copper in various conditions, copper and phosphor bronze specimens were included in this series of tests for comparative purposes.

Test results are listed in Table XV. For all materials, the corrosion rate at 800 ft from the ocean is only slightly more than one half that measured at 80 ft. At both locations, beryllium copper provides substantially better resistance to atmospheric corrosion than either copper or phosphor bronze, but all materials were covered with tarnish ranging in color from green to brown. Although temper or condition exerted no observable influence upon corrosion rate, beryllium copper was found to be slightly superior to beryllium-cobalt-copper. The changes noted in tensile properties were not consistent, and no correlation was found to exist between tension and weight loss data.

Table XVI provides a means of evaluating the present results in light of earlier work at several locations on the comparison materials copper and phosphor bronze. The higher corrosion rates obtained in the present study are probably due to the shorter exposure period, since Table XV indicates a marked drop in corrosion rate with increased time of test.

TABLE XVI.—COMPARISON OF SEVERAL INVESTIGATIONS OF CORROSION RATES IN MARINE ATMOSPHERES (STRIP OR SHEET SPECIMENS).

Material	Investigation	Nominal Composition, per cent[a]	Condition	Exposure Time, yr	Location	Corrosion Rate, mdd	Change, per cent	
							Tensile Strength	Elongation
Phosphorus-deoxidized copper	Present	0.025 P	A	2	Kure Beach, N. C.	0.4	+2.1	−7
	Tracy, Thompson, and Freeman (4)	0.020 P	A	8–10	Long Island Sound, Conn.	0.2	−1.6	−3
	ASTM (12)	0.007 P	A	10	Sandy Hook, N. J.	0.2	−1.4	−6
					Key West, Fla.	0.1	−1.0	−3
					La Jolla, Calif.	0.3	−0.6	+4
Phosphor bronze	Present	5.0 Sn, 0.016 P	A	2	Kure Beach, N. C.	0.5	+1.5	+2
	ASTM (12)	8.0 Sn, 0.03 P	A	10	Sandy Hook, N. J.	0.3	+1.3	0
					Key West, Fla.	0.2	+4.1	−2
					La Jolla, Calif.	0.5	+3.5	−1

[a] Balance copper.

CONCLUSIONS

Beryllium copper and beryllium-cobalt-copper in various tempers and conditions have been exposed to sea water and marine atmospheres. For comparative purposes, phosphorus deoxidized copper, arsenical admiralty, iron-modified 70-30 cupro-nickel, and phosphor bronze (Grade A) have been included in the annealed condition in many of these tests. The results over test periods up to 2 yr have been expressed as loss of weight or change in tensile properties.

In quiet sea water, the corrosion resistance of beryllium copper was less than that of cupro-nickel but equal to copper or admiralty. With increasing water velocities, the resistance offered by beryllium copper was still less than curpo-nickel but superior to copper or admiralty. In flowing sea water, partial immersion or "waterline effect" did not cause any appreciable change in corrosion rate.

High velocity tests, such as rotating-spindle, EES, and jet impingement yielded results that are slightly more anomalous. For example, beryllium copper offered relatively better resistance to corrosion in the rotating-spindle and EES tests than in the sea-water jet test. Galvanic potential and corrosion under open-circuit and polarizing conditions were similar to the values obtained with other copper-base materials.

Beryllium copper was found to offer better resistance to corrosion by marine atmospheres than either copper or phosphor bronze. As expected, the corrosion rate at 80 ft from the sea was substantially greater than encountered at 800 ft.

From the tests conducted, beryllium copper provides slightly better resistance to sea water or marine atmosphere than beryllium-cobalt-copper. Although cold rolling or precipitation hardening causes a marked improvement in the strength and hardness of these alloys, material condition exerts no observable influence upon corrosion resistance. The slight decrease in tensile strength and elongation resulting from edge corrosion does not appear significant. For all materials, the corrosion rate decreases with increasing exposure time but increases, often rapidly, with a rise in velocity. Although fair agreement has been observed between the present test results and similar work by earlier investigators, there are several instances where the data do not agree with service experience.

These tests indicate that the corrosion resistance of beryllium copper compares favorably with that of copper under many conditions of sea water or marine atmosphere exposure. Consequently, beryllium copper can be considered for marine applications requiring high strength and the corrosion resistance of copper.

Acknowledgment:

The assistance of The American Brass Co., and A. W. Tracy in particular, in furnishing the comparison materials is greatly appreciated. The author is especially indebted to F. L. LaQue and the staff of The International Nickel Co. for many helpful suggestions and for conducting these tests at Harbor Island and Kure Beach, N. C.

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DISCUSSION

MR. F. L. LAQUE[1] (*presented in written form*).—The data presented by Mr. Richards in this paper should provide a satisfactory picture of what may be expected of beryllium copper alloys in resisting deterioration in marine environments.

On the basis of the writer's experience with the relation of behavior under tests such as described to behavior in service, and particularly with respect to performance where the erosive effects of sea water at high velocity are encountered, the beryllium copper alloys can be rated as being superior to Admiralty brass and inferior to the iron modified 70-30 cupro-nickel alloy—as illustrated typically by the weight loss *versus* time curves in Fig. 2 in the paper and by the results of the erosion tests with the Engineering Experiment Station apparatus in Table XI. Less significance should be attached to the results of the jet impingement tests. A review of a mass of data from such tests as ordinarily conducted at Harbor Island and comparison with other tests and practical experience have led to the conclusion that this jet test is too drastic. It causes appreciable attack of many alloys that resist erosion very well in other tests and in service and tends to crowd the better materials into a narrow range of performance so that important differences in resistance to impingement attack observed in other ways are masked in the test.

While the polarization data in Fig. 8 support the view that beryllium copper will act like many other copper-base alloys in galvanic couples with less noble materials, they are not particularly helpful in interpreting the relationships between corrosion rates and potentials shown in Table XIV. The latter are of more interest as separate items of information than in establishing any instructive relationship between potentials and corrosion rates.

It should not be concluded from the data in Table XIV that the low (0.8 per cent) iron content 90-10 cupro-nickel alloy will always demonstrate a more noble potential and lower rate of corrosion in flowing sea water than a similar alloy of higher iron content.

MR. A. W. TRACY.[2]—I should like to emphasize two items in Mr. Richards' paper. The first concerns the higher corrosion resistance of the heat-hardened beryllium copper alloy specimens. Some time ago we made tests exposing beryllium copper to a jet of sea salt solution at a velocity of 36 ft per sec and also found that the material in the heat-hardened condition was somewhat more resistant to corrosion than material in the solid solution condition. We have obtained the same results on other materials that can be hardened by a change in structure. It may be that more permanent films of corrosion products can form under conditions of impingement on the hardened materials because they are more resistant to minute amounts of deformation.

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[1] In charge, Corrosion Engineering Section, Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[2] Assistant Metallurgist, The American Brass Co., Waterbury, Conn.

I should also like to add emphasis to Mr. Richards' statement that the data in his sea water tests do not always check the corrosion data on condenser tubes under actual operating conditions. Some of his data indicate a low resistance to impingement corrosion by 70-30 cupro-nickel. On the basis of experience, I would rate the resistance of this alloy to corrosion by turbulently flowing sea water to be definitely greater than that of either copper or beryllium copper.

MR. J. T. RICHARDS (*author's closure*). —The author would like to thank the discussers for their interesting contributions. Mr. LaQue's evaluation of the jet impingement tests is especially helpful, since the high velocity results appear more uniform when the jet data are omitted.

Mr. Tracy has called attention to the higher corrosion resistance of the precipitation-hardened beryllium copper. At first glance, this situation appears anomolous, since it would normally be expected that precipitation of the gamma phase from the alpha matrix might lead to selective attack. In the fully-aged condition, however, actual precipitation has either not occurred or is submicroscopic in size. Over-aging would, of course, produce gamma precipitate visible under the microscope, but it is probable that this high-beryllium phase (approximately 12 per cent beryllium) offers excellent corrosion resistance due to its enhanced ability to form a refractory film of beryllium oxide.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EFFECT OF TEMPERATURE ON THE BRITTLE BEHAVIOR OF METALS, WITH PARTICULAR REFERENCE TO LOW TEMPERATURES

The Symposium on Effect of Temperature on the Brittle Behavior of Metals was held under the auspices of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals during the First, Second, Third, Fifth, and Sixth Sessions of the Fifty-sixth Annual Meeting of the Society in Atlantic City, N. J., June 28–30, 1953. The purpose of this symposium is to bring together and summarize the existing knowledge of metallic materials at low temperatures in an effort to make designers and materials engineers more fully cognizant of the joint rôle played by metallurgical and mechanical factors and their influence on the behavior of metals at low temperatures. The papers presented in these five sessions encompass the following subjects: the evaluation of brittle failures in ships and engineering structures; criteria of metal behavior for design engineers; metallurgical and mechanical factors; significance and reliability of notch toughness tests; and certain aspects of current research, including new data on titanium.

The following papers were presented:

Introduction—A. L. Tarr
Brittle Failure in Ships and Other Steel Structures—K. K. Cowart
Analysis of Brittle Behavior in Ship Plates—M. L. Williams
A Critical Survey of Brittle Fracture of Carbon Plate Steel Structures Other than Ships—M. E. Shank
Interest of the Army in Brittle Failures—T. T. Paul
Theory of Brittle Fracture and Criteria for Behavior at Low Temperatures—E. R. Parker
Brittle Fracture—S. L. Hoyt
Metallurgical Aspects of Low-Temperature Behavior in Ferrous Materials—C. H. Lorig
Fundamentals of Fracture in Metals—M. Gensamer
The Effects of Size Upon Fracturing—G. R. Irwin
Brittleness, Triaxiality, and Localization—W. P. Roop
Effect of Metallurgical Structures on the Impact Properties of Steels—J. A. Rinebolt
Evaluation of the Significance of Charpy Tests—William S. Pellini
Significance of V-Notched Impact Test in Evaluation of Armor Plate—A. Hurlich
Notch Bend Tests for Evaluating the Properties of Weldments—R. D. Stout
Reproducibility of Keyhole Charpy and Tear-Test Data on Laboratory Heats of Semikilled Steels—R. H. Frazier, J. W. Spretnak, and F. W. Boulger
Effect of Specimen Preparation on Notch-Toughness Behavior of Keyhole Charpy Specimens in the Transition Temperature Zone—R. W. Vanderbeck, R. W. Lindsay, H. D. Wilde, W. T. Lankford, and S. C. Snyder

High Speed Motion Picture Study of Impact Test—H. L. Fry
Low Temperature Impact Properties of Titanium—David E. Driscoll
Effect of Impact Properties of Quenched and Tempered Steels—H. Schwartzbart and J. P. Sheehan
The Notched Bar Impact Properties of Tempered Martensite in Medium Carbon, Medium Alloy Grades of Steel—M. Baeyertz, W. F. Craig, Jr., and J. P. Sheehan
Brittleness of Steels at High Hardness Levels—Sam Tour
Notch Sensitivity of Steels—E. J. Ripling
Effect of Carbon and Nitrogen on the Tensile Deformation of High-Purity Iron at 27 C and at −196 C—Lewis D. Hall
Tension Impact Strength and Strain Distribution at Room and Sub-Zero Temperatures of Stainless and Other Steels—C. R. Mayne, V. N. Krivobok, and C. W. Muhlenbruch
Low Temperature Toughness of Flake and Spheroidal Graphite Cast Irons—J. S. Vanick
Ductile and Brittle Failure in Ferritic Nodular Iron—G. N. J. Gilbert
The Low-Temperature Properties of Cast Iron—G. N. J. Gilbert
Impact Properties of Ferritic Ductile Iron—R. W. Kraft

These papers together with discussions are issued as *ASTM Special Technical Publication No. 158* entitled "Symposium on Effect of Temperature on the Brittle Behavior of Metals with Particular Reference to Low Temperatures."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TECHNIQUES FOR ELECTRON METALLOGRAPHY

At the Thirteenth and Seventeenth Sessions of the Fifty-sixth Annual Meeting of the American Society for Testing Materials held on June 30, 1953, in Atlantic City, N. J., Committee E-4 on Metallography sponsored a Symposium on Techniques for Electron Metallography.

The purpose of this symposium was to present examples of techniques in common use or of potential value, in the hope that increased interest in the application of electron microscopy to metallurgical problems will result.

The following papers,[1] with their discussions, are issued as *ASTM Special Technical Publication No. 155* entitled "Symposium on Techniques for Electron Metallography."

Introduction—C. M. Schwartz
Techniques Used in Electron Microscopy of Aluminum Alloys—M. S. Hunter and F. Keller
Techniques for the Study of Precipitated Carbides—W. D. Forgeng and John L. Lamont
Inorganic Replication: Interpretation of Electron Micrographs—C. J. Calbick
Electron Microstructure of Steel by "Extraction Replica" Technique—R. M. Fisher
Metal Shadowing for Contrast Enhancement—Comparison of Shadow Metal and Shadow Angle—D. M. Teague
Specimen Polishing Techniques for Electron Metallography of Steel—W. L. Grube and S. R. Rouze
Techniques That Permit Successive Examination of Specific Areas by Electron Microscopy—T. A. McLauchlin
Replica Washing Methods—Ernest F. Fullam
Application of New Etchants for Delineation of Fine Structure in Steel—A. E. Austin
Positive *versus* Negative Plastic Replicas—C. M. Schwartz

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[1] Another paper entitled "Determination of Surface Properties for Eutectoid Steel and Iron as Prepared by the Conventional Metallographic Techniques of Abrading, Mechanical Polishing, and Chemical Etching," by C. F Tufts was presented at this Symposium and will be published separately at a later date.

SOME EFFECTS OF THE GRADING OF SAND ON MASONRY MORTAR*

By C. C. Connor[1]

Synopsis

The effect of 22 masonry sands upon the workability of their mortars and the influence of 13 of these sands upon the amount of cracking that occurred between brick and mortar at existing buildings were studied. All of the mortars were high-lime mortar mixes, and the measurements of mortar joint cracking were made at building units where the conditions were uniformly favorable to low percentages of joint cracking. The data indicated that a sand, to produce highly workable mortars, should have sieve fractions which increase progressively by liberal amounts from coarse to fine up to and including the No. 30 to No. 50 sieve fraction, an amount retained on the No. 16 sieve less than 25 per cent, an amount retained on the No. 30 sieve less than 50 per cent, and no excessive concentration of material passing the No. 50 sieve. The percentages of mortar joint cracking varied widely. Low percentages occurred consistently where the sands produced highly workable mortars and had less than 25 per cent passing the No. 50 sieve.

Grading requirements are suggested for masonry sands based upon the percentages in the various sieve fractions and upon limitations in certain fractional divisions. These suggested requirements apparently provide an improved method of judging the suitability of sands for masonry mortar.

Every competent mason knows that the quality of the sand exerts an important influence upon the workability of the mortar. Most masons have struggled with mortars made harsh and unworkable by poor sands, and have experienced the change in the same mortar mixes to smooth and workable mortars when a sand with workable characteristics was used. But neither the mason nor the engineer has any means of judging with certainty what the effect a given sand will have on the workability of a mortar mix, nor can either consistently select the best sand for workability from the samples of several. Moreover, there is a growing amount of evidence that certain sands which produce mortars with excellent workability in good mortar mixes are associated with high percentages of mortar joint cracking. Unquestionably, a good mortar must have the capacity to form complete bonds with the masonry units and maintain those bonds for the life of the masonry.

It has been widely recognized that the grading of the aggregate has an effect upon workability. Furnas (1)[2] pointed out the importance of the relationship between workability and the grading of concrete aggregates and suggested that

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Building Inspection and Maintenance Engineer, New Jersey Bell Telephone Co., Newark, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 945.

"particles of the next size smaller than the largest may be expected to act as ball bearings, and so on down the line of sizes." Anderegg (2) stated that "workability depends upon the water and the grading of the whole system, including the cementing material." He also stated, "the workability depends upon the presence of a sufficient number of ball bearings of suitable size distribution."

The purpose of this investigation was to study various masonry sands and their mortars and, if possible, to determine the following:

1. The relationship, if any, of the sieve analysis of the sand to the workability of the mortar.
2. The relationship, if any, of the sieve analysis of the sand to the amount of cracking that occurs between the brick and the mortar joints in existing buildings after a period of exposure.
3. The sieve analysis requirements for sands which, in a good mortar mix, would produce mortars with good workability and with the capacity to form complete and durable bonds with brick having properties favorable to such bonds.

This investigation is an outgrowth of two previous field studies (3, 4). One of these studies covered the separation cracking or mortar joint cracking which occurred in existing buildings; the other covered factors in the moisture resistance of brick masonry walls. It was established in those studies that the amount of mortar joint cracking was closely associated with leaks in brick masonry walls. Sand did not appear as a definite factor in moisture resistance because good sands were used in the great majority of the building units studied. However, the possible influence of sand was commented upon.

In recent years a considerable number of different sands have been used in the building units constructed in the continuation of the test program of buildings specially designed for moisture resistance, the first part of which was reported on in one of the previous papers (4). It soon became evident that the various sands affected the workability of the mortars and also that they had a very considerable influence upon the amount of mortar joint cracking that occurred. Since the first test building, a total of 113 building units were constructed during the fourteen year period up to 1952. Only five have leaked, but the leaks at three units were apparently attributable to the sand used; and the sand was a contributing factor in the leaks at another. The higher percentages of mortar joint cracking apparently caused by poor sands at a number of other building units further indicated the hazard of their continued use.

Sands Studied:

Twenty-two widely used masonry sands were included in the study. All were natural sands from pits, and all were washed except one, sand No. 9.

Sieve analyses were made of all sands in accordance with the ASTM standard method of test.[3] Some of the sieve analyses were made by commercial laboratories on submitted samples, and others were made by the author on job samples or samples from the sand pits. Sand No. 6 was a Potomac River sand used in tests at the National Bureau of Standards. Its sieve analyses were taken from various published papers (5 to 8) where the analyses were made in the course of the tests reported upon. The sieve analysis for each sand as given in Table II is the average of all available sieve analyses for that particular sand where reported in terms of the present standard set of sieves. The sand was analyzed during construction for only 14 of the 26 building units studied.

No attempt was made to judge the shape of the particles in the various sands, but all were somewhat angular.

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[3] Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46), 1952 Book of ASTM Standards, Part 3, p. 954.

MORTARS STUDIED

There were four mortar mixes included in the study—all high-lime mortars. Measurements of the mortar joint cracking associated with the different mortars were made at existing building units. The mortar mix at 22 of the building units was 1:1:5 by volume; at two units the mortar mix was 1:1:6; at one unit the mortar mix was 1:1:4.8, the damp sand volume being increased to equal 4.8 volumes of dry sand; and at one building unit the mortar mix was 1.3:1.5:7. The latter was a masonry cement mortar composed of cement, normally hydrated lime, and sand. The composition of the masonry cement given is according to information from the manufacturer. In all mortars the volumes of sand were measured in a damp condition as the sand came from piles at the job, but the one noted was corrected to dry sand volumes.

Types of Lime Used in the Mortars Studied:

The lime in the mortar at three of the building units where cracking measurements were made was a normal hydrate. At 22 of the building units the lime was a special hydrate, and at one building unit the lime was a quicklime putty. All limes, except the quicklime putty, were added to the mortar as a dry powder.

The plasticity of none of the limes was measured at the time of use, but tests at various times showed that all of the special hydrates used had plasticity measurements that exceeded 300 when tested in accordance with the ASTM standard method of test.[4] The plasticity of the quicklime putty was apparently high, and the plasticity of the normal hydrates was probably above average for that type of lime. The workability of the mortars as judged by masons indicated that the plasticity of the limes used was good in all cases.

Method of Testing Mortars:

All mortars were judged as to their workability by masons on the job with the exception of the mortar produced by sand No. 6. The judgments of the masons were made on the basis of the trowelability, cohesiveness, and the stickiness of the mortars. Trowelability was judged by noting the ease with which the mortars spread and tooled. Cohesiveness was judged on the basis of the capacity of the mortars to be spread out with the point of the trowel without breaking or separating. The stickiness of the mortars was judged from the adhesiveness of a thick buttering on the end of a brick and the manner in which the mortar separated from the trowel. A mortar had to be satisfactory in all of these tests to be given an excellent rating for workability. The rating generally was a composite opinion of a number of masons. In several instances, comparative tests were made of different available sands by a master mason. In two cases, the mortar with a rejected sand was fattened up by reducing the amount of sand in the mix until it became passably workable. The mortar mix at that point was recorded as experimental information.

Sand No. 6, the Potomac River sand used in tests at the National Bureau of Standards, was considered to produce a high-lime mortar with excellent workability, if the lime were plastic, on the basis of the information given in the papers reporting those tests (**5** to **11**).

Measurement of Mortar Joint Cracking at Existing Buildings:

The measurements of mortar joint cracking, or separation cracking, were made at 46 building units, but the results at only 26 are reported. The age of the various units ranged from 2 to 24 years.

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[4] Standard Method of Physical Testing of Quicklime and Hydrated Lime (C 110 - 49), 1952 Book of ASTM Standards, Part 3, p. 234.

TABLE I.—WORKABILITY OF MORTAR WITH VARIOUS SANDS AND ASSOCIATED MORTAR JOINT CRACKING.

Sand	Classification Of Sand	Building Unit Number	Building Age, yr	Nominal Mortar Mix (Volume)	Type of Lime[b]	Mortar Workability Judged by Masons	Average Mortar Joint Cracking Under Favorable Conditions[a]	
							With Smooth Surfaced Brick, per cent	With Rough Surfaced Brick, per cent
No. 1	Good	1	24	1.3:1.5:7	NH	Excellent		4.8
		2	22	1:1:6	NH	Excellent	6.0	
		3	22	1:1:6	NH	Excellent	12.3	
							Avg. 9.1	Avg. 4.8
No. 2	Good	4	5	1:1:5	SH	Excellent	11.0	
No. 3	Good	5	4	1:1:5	SH	Excellent	9.6	
		6	4	1:1:5	SH	Excellent	10.8	
		7	15	1:1:4.8[c]	Q	Excellent	10.6	
							Avg. 10.3	
No. 4	Good	8 A	11	1:1:5	SH	Excellent		9.4
		8 B	11	1:1:5	SH	Excellent	9.0	
No. 5	Good	9	3	1:1:5	SH	Excellent		7.1
No. 6	Good[d]	...	...	...	...	Excellent[d]	...	...
No. 7	Good	10	4	1:1:5	SH	Excellent		4.5
No. 8	Good	11	2	1:1:5	SH	Excellent	11.3[e]	
No. 9	Workable			1:1:5	SH	Good	[f]	
No. 10	Workable			1:1:5	SH	Excellent	[f]	
No. 11	Workable			1:1:5	SH	Excellent	[f]	
No 12	Workable			1:1:5	SH	Excellent	[f]	
No. 13	Workable			1:1:5	SH	Excellent	[f]	
No. 14	Workable			1:1:5	SH	Fair	[g]	
No. 15	Unsatisfactory[h]	12	5	1:1:5	SH	Excellent	22.9	
		13	4	1:1:5	SH	Excellent	38.1	
		14	5	1:1:5	SH	Excellent		5.3
		15	4	1:1:5	SH	Excellent		8.1
		16	4	1:1:5	SH	Excellent		14.2
							Avg. 30.5	Avg. 9.2
No. 16	Unsatisfactory[h]	17	4	1:1:5	SH	Excellent	40.7	
		18	3	1:1:5	SH	Excellent	20.7	
							Avg. 30.7	
No. 17	Unsatisfactory[h]	19	5	1:1:5	SH	Excellent	56.5	
No. 18	Unsatisfactory[i]	20 A	11	1:1:5	SH	Poor	14.8	
		20 B	11	1:1:5	SH	Poor	36.0	
							Avg. 25.4	
No. 19	Unsatisfactory[i]	21	11	1:1:5	SH	Fair[j]		7.0
		22	5	1:1:5	SH	Poor		15.0
		23	11	1:1:5	SH	Poor	26.0	
							Avg. 26.0	Avg. 11.0
No. 20	Unsatisfactory[i]	24	4	1:1:5[k]	SH	Poor	18.9[l]	
No. 21	Unsatisfactory	...	...	1:1:5[m]	SH	Poor	...	
No. 22	Unsatisfactory	...	...	1:1:5[n]	SH	Poor	...	

[a] Brick with rate of absorption between 5 g and 25 g in 1 min and with no loosely sanded surfaces; high-lime mortar containing lime of good plasticity and showing no evidence of over sanding; concave tooled mortar joints; workmanship apparently good.

[b] Type of lime: NH–normal hydrate; SH–special hydrate; Q–quicklime.

[c] Volumes of dry sand.

[d] Potomac River sand. Gave good results and excellent workability in high-lime mortars containing plastic limes in tests at the National Bureau of Standards. No mortar joint cracking measurements.

[e] With loosely sanded brick. Lowest cracking with this unfavorable condition.

[f] No mortar joint cracking record under favorable conditions in buildings two years old or more.

[g] Tested by master mason but not used.

[h] Produced mortars with excellent workability but the associated mortar joint cracking was high.

[i] Poor workability mortar unless fattened, and high mortar joint cracking.

[j] Mortar probably fattened.

[k] Mortar had to be fattened to a 1:1:4.4 mix to give it good workability.

[l] Mortar joint cracking with a loosely sanded brick and a fattened mortar. Cracking 38.3 per cent with the same brick and a slightly oversanded mortar.

[m] Poor workability mortar. Had to be fattened to a 1:1:4 mix to make it passably workable. Rejected for use.

[n] Poor workability mortar. Had to be fattened to a 1:1:3.5 mix to make it passably workable. Rejected for use.

At some of the buildings, different walls had face brick with dissimilar characteristics, and these were classed as separate building units. All walls were integral and 12 in. thick. All of the building units were subject to the severe exposure characteristic of a seashore state, and the range of temperature was wide.

It has been established that the amount of mortar joint cracking is influenced by characteristics of the mortar as well as the brick (**3**, **4**). The amount of mortar joint cracking associated with the various sands, therefore, was used as a measure of the suitability of the sands for masonry mortar.

The gathering of truly significant data from the field to evaluate the influence of one particular factor is difficult because of the number of factors involved in the construction of brickwork. To compare properly the effect of different sands upon mortar joint cracking, it was necessary that all of the other factors be the same, or at least uniformly favorable to low percentages of cracking. Therefore, all the measurements from building units where any known unfavorable factor occurred were eliminated from statistical consideration, except in two cases as noted.

The following conditions were considered unfavorable to low percentages of mortar joint cracking: Brick with a rate of absorption above or below the range of 5 g to 25 g of water absorbed in 1 min. when the dried brick was set flat side down in $\frac{1}{8}$ in. of water; mortar joint tooling other than concave; brick with loosely sanded surfaces; oversanded mortar; and poor workmanship. Evidence that the first two conditions listed increased the mortar joint cracking has been presented (**3**, **4**), and a considerable number of measurements have indicated that the other conditions have a similar unfavorable effect.

Twenty-four of the building units were free from conditions unfavorable to low mortar joint cracking; but measurements were reported from two additional building units where the brick had loosely sanded surfaces. These measurements were significant for two sands, and none was available under uniformly favorable conditions.

All the brick in the units had been tested for initial rate of absorption according to the ASTM standard method of tests[5] except at five units where the rate of absorption had been tested at the exposed face of the brick by the method described previously (**3**).

Method of Measuring Mortar Joint Cracking:

All measurements of mortar joint cracking were made in selected panels of brickwork and in the same manner as described previously (**3**). The measurements were made with a tapeline and are approximate only. However, repeated measurements did not vary more than 10 per cent.

DATA

Table I gives all the data for the study except the sieve analyses of the various sands which are given in Table II. The good sands are those which in field use had produced mortars with excellent workability as judged by masons, and were consistently associated with low percentages of mortar joint cracking where all the other conditions were favorable. The joint cracking accompanying sand No. 8 may have been adversely affected by the loosely sanded surfaces of the brick, but the percentage was low. Sand No. 6, the Potomac River sand was included in this group, although it was not used or observed by the author. In the masonry research at the National

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[5] **Standard Methods of Sampling and Testing Brick (C 67 – 50), 1952 Book of ASTM Standards, Part 3, p. 414.**

Bureau of Standards (**5** to **11**) it was judged workable, or produced high flow after suction, in high-lime mortars containing plastic lime, and produced some very impenetrable masonry assemblages.

TABLE II.—SIEVE ANALYSES OF VARIOUS SANDS WITH ASSOCIATED MORTAR JOINT CRACKING.

Sand	Sieve Analyses Fractions, per cent							Fineness Modulus	Sieve Analyses Averaged	Workability in High-Lime Mortar Judged by Masons	Average Mortar Joint Cracking Under Favorable Conditions[a]	
	On No. 4	No. 4–8	No. 8–16	No. 16–30	No. 30–50	No. 50–100	Passing No. 100				With Smooth Surfaced Brick, per cent	With Rough Surfaced Brick, per cent
Good Sands[b]												
No. 1......	0.0	4.0	17.1	23.1	38.7	14.5	2.6	2.50	1	Excellent	9.1	4.8
No. 2......	0.1	2.0	12.4	30.0	41.1	12.1	2.3	2.45	3	Excellent	11.0	
No. 3......	0.0	2.5	9.3	28.2	52.0	5.5	2.5	2.44	1	Excellent	10.3	
No. 4......	0.0	0.2	6.9	24.3	51.5	14.5	2.6	2.19	3	Excellent	9.0	9.4
No. 5......	0.0	0.3	9.5	27.5	42.0	17.9	2.8	2.24	1	Excellent		7.1
No. 6[c].....	0.0	0.2	4.4	18.0	57.7	16.7	3.0	2.05	4	Excellent[c]		
No. 7......	0.0	0.2	4.8	23.6	47.8	21.4	2.2	2.08	1	Excellent		4.5
No. 8......	0.0	0.8	7.8	29.8	43.4	14.3	3.9	2.26	3	Excellent	11.3[d]	
											Avg. 10.1	Avg. 6.5
Workable Sands[e]												
No. 9......	0.7	3.2	9.4	26.4	42.9	15.2	2.2	2.38	3	Good		
No. 10.....	0.0	0.0	7.0	26.8	47.8	16.3	2.1	2.20	1	Excellent		
No. 11.....	0.0	0.2	7.9	32.1	42.5	15.0	2.3	2.29	3	Excellent		
No. 12.....	0.0	0.1	3.3	30.6	50.0	14.4	1.6	2.20	2	Excellent		
No. 13.....	0.1	3.2	10.0	21.4	43.3	18.6	3.4	2.26	3	Excellent		
No. 14.....	0.0	3.2	11.9	31.7	34.5	13.1	5.6	2.41	1	Fair		
Unsatisfactory Sands[f]												
No. 15[g]....	0.1	0.8	8.1	21.0	30.4	30.3	9.3	1.91	4	Excellent	30.5	9.2
No. 16[g]....	0.0	1.4	10.5	21.0	30.4	24.6	12.1	1.97	4	Excellent	30.7	
No. 17[g]....	0.4	2.6	4.8	18.4	45.7	23.8	4.3	1.95	1	Excellent	56.5	
No. 18.....	0.0	4.8	14.9	29.6	25.0	16.2	9.5	2.39	1	Poor	25.4	
No. 19.....	1.2	4.7	10.2	52.0	24.3	5.7	1.9	2.82	1	Poor	26.0[h]	11.0
No. 20.....	0.0	0.5	18.9	32.3	36.1	10.8	1.4	2.58	2	Poor		
No. 21[i]....	0.0	1.2	9.1	52.5	28.3	5.9	3.0	2.62	1	Poor		
No. 22[i]....	0.0	0.0	0.1	0.4	18.4	56.0	25.1	0.94	1	Poor		
											Avg. 33.8	Avg. 10.1

[a] Brick with rate of absorption between 5 g and 25 g in 1 min and with no loosely sanded surfaces; high-lime mortar containing lime of good plasticity and showing no evidence of oversanding; concave tooled mortar joints; workmanship apparently good.

[b] Produced workable mortars and low joint cracking in field use.

[c] Potomac River sand. Gave good results and excellent workability in high-lime mortars containing plastic limes in tests at the National Bureau of Standards. No mortar joint cracking measurements.

[d] With loosely sanded brick. Lowest cracking with this unfavorable condition.

[e] Produced mortars with good workability, but not used under favorable conditions at buildings with an exposure period long enough to insure significant mortar joint cracking measurements.

[f] High mortar joint cracking, or poor workability, or both.

[g] Produced mortars with excellent workability, but the associated mortar joint cracking was high.

[h] Mortar joint cracking with loosely sanded brick and a fattened mortar was 18.9 per cent. Cracking was 38.3 per cent with the same brick and a slightly oversanded mortar.

[i] Produced mortar of poor workability unless excessively fattened. Rejected for use.

Many of the test assemblages were very resistant to water penetration after 1 to 3 years' exposure (**10**), indicating that low percentages of mortar joint cracking were probable.

The workable sands were those which produced mortars with workabilities judged to be fair, good, or excellent. None of these sands was used in building units which had exposure periods of as much as two years where the conditions were uniformly favorable. Sand No. 14 was tested but not used.

The unsatisfactory sands produced mortars of poor workability or were associated with high percentages of mortar joint cracking. Sands Nos. 18, 19, and 20 were unsatisfactory on both counts.

Table II gives the sieve analyses of the various sands, their fineness moduli, the workability of their mortars, and the associated mortar joint cracking with smooth surfaced and rough surfaced brick.

Table III gives the relationship of the fraction between the Nos. 30 and 50 sieves to the fraction between the Nos. 16 and 30 sieves for the various sands. It also gives the percentages retained on the No. 16 and on the No. 30 sieves, and the percentage passing the No. 50 sieve to show the amount of very coarse, coarse, and very fine particles in each sand. The fineness moduli, the workability produced by the various sands in mortars, and the associated mortar joint cracking are repeated for easy reference.

TABLE III.—SIGNIFICANT SAND GRADING RELATIONSHIPS AND DIVISIONS WITH ASSOCIATED MORTAR JOINT CRACKING.

Sand	Sieve Analysis Fractions, per cent				Fineness Modulus	Workability in High-Lime Mortar Judged by Masons	Average Mortar Joint Cracking Under Favorable Conditions[a]	
	Excess of the No. 30–50 Fraction over the No. 16–30 Fraction	Retained on No. 16	Retained on No. 30	Passing No. 50			With Smooth Surfaced Brick, per cent	With Rough Surfaced Brick, per cent
Good Sands[b]								
No. 1	67.5	21.1	44.2	17.1	2.50	Excellent	9.1	4.8
No. 2	37.0	14.5	44.5	14.4	2.45	Excellent	11.0	
No. 3	84.4	11.8	40.0	8.0	2.44	Excellent	10.3	
No. 4	111.9	7.1	31.4	17.1	2.19	Excellent	9.0	9.4
No. 5	52.7	9.8	37.3	20.7	2.24	Excellent		7.1
No. 6[c]	220.6	4.6	22.6	19.7	2.05	Excellent		
No. 7	102.5	5.0	28.6	23.6	2.08	Excellent		4.5
No. 8	45.6	8.6	38.4	18.2	2.26	Excellent	11.3[d]	
							Avg. 10.1	Avg. 6.5
Workable Sands[e]								
No. 9	62.5	13.3	39.7	17.4	2.38	Good		
No. 10	78.4	7.0	33.8	18.4	2.20	Excellent		
No. 11	32.4	8.1	40.2	17.3	2.29	Excellent		
No. 12	63.4	3.4	34.0	16.0	2.20	Excellent		
No. 13	102.3	13.3	34.7	22.0	2.26	Excellent		
No. 14	8.8	15.1	46.8	18.7	2.41	Fair		
Unsatisfactory Sands[f]								
No. 15[g]	44.8	9.0	30.0	39.6	1.91	Excellent	30.5	9.2
No. 16[g]	44.8	11.9	32.9	36.7	1.97	Excellent	30.7	
No. 17[g]	148.4	7.8	26.2	28.1	1.95	Excellent	56.5	
No. 18	15.5 less	19.7	49.3	25.7	2.39	Poor	25.4	
No. 19	53.3 less	16.1	68.1	7.6	2.82	Poor	26.0[h]	11.0
No. 20	11.8	19.4	51.7	12.2	2.58	Poor		
No. 21[i]	46.1 less	10.3	62.8	8.9	2.62	Poor		
No. 22[i]	450.0	0.1	0.5	81.1	0.94	Poor		
							Avg. 33.8	Avg. 10.1

[a] Brick with rate of absorption between 5 g and 25 g in 1 min and with no loosely sanded surfaces; high-lime mortar containing lime of good plasticity and showing no evidence of oversanding; concave tooled mortar joints; workmanship apparently good.

[b] Produced workable mortars and low joint cracking in field use.

[c] Potomac River sand. Gave good results and excellent workability in high-lime mortars containing plastic limes in tests at the National Bureau of Standards. No mortar joint cracking measurements.

[d] With loosely sanded brick. Lowest cracking with this unfavorable condition.

[e] Produced mortars with good workability, but not used under favorable conditions at buildings with an exposure period long enough to insure significant mortar joint cracking measurements.

[f] High mortar joint cracking, or poor workability, or both.

[g] Produced mortars with excellent workability, but the associated mortar joint cracking was high.

[h] Mortar joint cracking with loosely sanded brick and a fattened mortar was 18.9 per cent. Cracking was 38.3 per cent with the same brick and a slightly oversanded mortar.

[i] Produced mortar of poor workability unless excessively fattened. Rejected for use.

DISCUSSION OF DATA

Influence of Sand Grading Upon Mortar Workability:

Sands Nos. 1 to 17 inclusive produced workable mortars. The most obvious fact in the sieve analyses of these sands is that each fractional division between sieves is exceeded by the next finer sieve fraction down to and including the fraction between the No. 30 and No. 50 sieves. In the five unsatisfactory sands which failed to produce workable mortars, the increase in the amount of the fractions stopped short in three cases at the fraction between the Nos. 16 and 30 sieves. Apparently there were different causes for the lack of workability in sands Nos. 20 and 22, as will be discussed later. The progressive increase of the fractional divisions between sieves down to and including the No. 30 to No. 50 fraction appears to be the primary factor in the grading of the sands that produced workable mortars.

This progressive increase in fractional divisions is what might be expected from a system of roller bearings where theoretically each large particle in the No. 8 to No. 16 sieve fraction would be surrounded by particles from the next finer sieve fraction, and these in turn would each be surrounded by the still smaller particles from the No. 30 to No. 50 sieve fraction. The diminishing amount passing the No. 50 sieve indicates that these particles were not primary roller bearings in the system although they probably had some roller bearing effect. Apparently they also acted as void fillers. It is not likely that a theoretically perfect roller bearing system ever exists in an ordinary mortar mix.

Relationship of the No. 30 to No. 50 Sieve Fraction to the Next Coarser Fraction:

Just how much each sieve fraction should exceed the next coarser fraction in a workable sand is a difficult question, and apparently there are other factors in the sand grading that exert an influence on workability.

Sands Nos. 14 and 20 help to establish some of the limits in satisfactory grading. Sand No. 14 was a borderline sand. It was fairly workable, but produced a mortar of the stiffer type needed for very thick joints and developed considerably less workability in a mason's test than sands Nos. 13 and 15. Sand No. 20 is definitely on the unsatisfactory side. Its mortar had to be fattened by reducing the amount of sand to a 1:1:4.4 mix to produce good workability. In both these sands the sieve fractions increased steadily, and the fraction between the No. 30 and the No. 50 sieves was larger than the next coarser fraction, but by small percentages. The No. 30 to No. 50 sieve fraction in sand No. 14 exceeded the next coarser fraction by only 8.8 per cent, and in sand No. 20 the excess was only 11.8 per cent. The next lowest percentage of excess in the very workable sands was 32.4 per cent, and the percentages ranged upward to 220.6 per cent. A sand, to produce a highly workable mortar, apparently should have a No. 30 to No. 50 sieve fraction which exceeds the No. 16 to No. 30 sieve fraction by an amount considerably greater than the 11.8 per cent excess for sand No. 20.

Influence of the Amount of Coarse Material in the Sand on Mortar Workability:

The workability of the mortars produced by sands Nos. 14 and 20 seems to have been influenced also by the amount of coarse material retained on the No. 30 sieve. The 46.8 per cent for sand No. 14 and 51.7 per cent for sand No. 20, are higher than for any sand that produced highly workable mortars. Sand No. 14 squeezed by in a 1:1:5 mortar mix with fair workability, but the mortar with sand No. 20 apparently had particle interference unless the proportion of the cementitious material was increased by reducing the amount of sand in the mix.

The unsatisfactory sands Nos. 19 and 21 had 68.1 per cent and 62.8 per cent, respectively, retained on the No. 30 sieve. The mortar with sand No. 19 was rated as having fair workability at one building unit, but at two others the mortar workability was rated as poor. The mortar with sand No. 21 had to be fattened to a 1:1:4 mix before it could be troweled with reasonable facility. Both of these sands were very coarse, and their No. 30 to No. 50 sieve fraction was much smaller than the next coarser fraction. The result in a 1:1:5 mortar was poor workability for both sands. The unsatisfactory sand No. 18 had 49.3 per cent retained on the No. 30 sieve. It produced a mortar of poor workability, but it also had a deficiency in the No. 30 to No. 50 sieve fraction.

Sand No. 14, with 46.8 per cent, had the highest percentage retained on the No. 30 sieve of any sand which produced reasonably workable mortars. Judging from the percentages shown in Table III, it appears that the amount retained on the No. 30 sieve should not exceed 50 per cent if the sand is to produce a workable mortar. This estimated percentage may be too high, but if the No. 30 to No. 50 sieve fraction provides a liberal excess over the next coarser fraction, mortar workability might be achieved with this percentage, as is indicated by sand No. 2. This sand had 44.5 per cent retained on the No. 30 sieve, and the excess of the No. 30 to No. 50 sieve fraction over the next coarser fraction was 37.0 per cent. The workability of this mortar was one of the best, and apparently indicated that the amount of coarse material retained on the No. 30 sieve could be increased somewhat without material damage to the workability of the mortar.

There are no positive indications in the data regarding the maximum amount that can be retained on the No. 16 sieve without harm to workability. The largest amount retained was 21.1 per cent in sand No. 1, which produced mortars of excellent workability. However, the total of the percentages between sieves in a sieve analysis is inelastic, and it would be difficult to obtain a distribution of particle size which would permit the amount retained on the No. 16 sieve to exceed 25 per cent and retain the progressive increase in each sieve fraction up to and including the No. 30 to No. 50 sieve fraction with the percentages of increase in each fraction apparently required for mortar workability. And at the same time, the amount retained on the No. 30 sieve apparently should not exceed 50 per cent. It is generally conceded that a sand with too much very coarse material will not produce a mortar with good workability. In the grading requirements suggested later in Table IV, the maximum amount permitted to be retained on the No. 16 sieve is 25 per cent. This is admittedly an estimate not fully supported by the data, but is probably close to the top limit for a good masonry sand.

Influence of an Excess of Fine Material on Workability:

It is apparent that a sand can have such an excess of fine material that it reduces the workability of the mortar. Sand No. 22 was such a sand, and the mortar with this sand had to be fattened to a 1:1:3.5 mix before it was usable. This sand resembled an asphalt sand, and was one of the type known to masons as "dead" sands. Its mortar was never better than passably workable even when fattened, although it has had considerable use in masonry construction. The amount passing the No. 50 sieve was 81.1 per cent which compares with 39.6 per cent, the next highest for any of the sands which produced workable mortars.

Influence of Sand Grading on Mortar Joint Cracking:

A major criterion of a good masonry mortar is its capacity to form and maintain complete bonds with the masonry units. An examination of the data in the tables shows that all of the low percentages of cracking under 12 per cent were associated with sands producing mortars with excellent workability. It is logical to assume that a mortar must be workable to produce the complete contact and high extent of bond necessary for durable bonds, and this is borne out by the data.

The fact, however, that a sand will produce a workable mortar with plastic cementitious material is not a positive indication that it will produce durable bonds, as illustrated by sands Nos. 15, 16, and 17. These sands were exceptional in the amount passing the No. 50 sieve, as shown in Table III. With smooth surfaced bricks they were consistently associated with high percentages of mortar joint cracking. The amount passing the No. 50 sieve in these sands varied from 28.1 to 39.6 per cent. These high percentages apparently contributed to the workability of the mortars, but the associated mortar joint cracking was a great deal higher than with any of the other sands which produced workable mortars. In the other workable sands, the highest percentage passing the No. 50 sieve was 23.6 per cent.

Sands Nos. 15, 16, and 17, under favorable conditions, showed no serious joint cracking in less than six months. The high percentages of cracking developed later at some unknown time. In two cases it was discovered in less than two years, but on two sheltered walls at building unit No. 17 it required over three years to develop. The apparent cause of the high percentages of mortar joint cracking was volume change subsequent to the hardening of the mortar. The increased number of cementitious coatings around the fine particles may have increased this type of volume change.

It may well be significant that concretes containing fine aggregate from the same source as the fine sand No. 16 have shown a decided tendency to surface craze and develop serious cracks in walls, slabs, and cement finish; while there has been a noteworthy minimum of cracks and crazing in concretes where the fine aggregates were from the same sources as the good sands Nos. 1, 2, 4, and 13.

Significant Comparisons of Mortar Joint Cracking Accompanying Good and Unsatisfactory Sands:

The best comparison between the various sand gradings is furnished by the average mortar joint cracking which occurred with smooth surfaced brick where all other conditions were favorable to low percentages of cracking. As shown in Tables II and III, the average joint cracking with smooth surfaced bricks for sands in the unsatisfactory group was 33.8 per cent as compared with the average cracking of 10.1 per cent accompanying the good sands. It seems significant that the differences in mortar joint cracking occurred consistently with certain obvious differences in the grading of the sand.

Rough surfaces on brick evidently exerted considerable restraint on the development of mortar joint cracking, but the average cracking was considerably lower with good sands than with unsatisfactory sands. It is doubted, however, that this is necessarily significant.

While the mortar joint cracking at 20 buildings was not reported because other factors increased the cracking and obscured the effect of the sand, the average joint cracking with unsatisfactory sands was 27.8 per cent, which compares with 19.9 per cent for the good sands.

Perhaps the best illustration of the detrimental effect of an unsatisfactory sand grading was the difference in mortar joint cracking at building units Nos. 8B and 20B where all conditions were practically identical except for the sand used. The mortars were the same mix and contained the same brand of pressure hydrated lime. The bricks were identical in make and tested almost the same for rate of absorption. At building unit No. 8B, sand No. 4 was used, and the mortar joint cracking was 9.0 per cent. At building unit No. 20B, the unsatisfactory sand No. 18 was used, and the mortar joint cracking was 36.0 per cent, or four times as great. Workmanship was approximately equal, and in both cases the walls were sheltered court walls. There was no apparent explanation for the great difference in joint cracking except the grading of the sand.

Fineness Modulus:

The fineness modulus for each of the various sands has been given in Tables II and III as a matter of information. Several of the best sands have a fineness modulus lower than generally regarded as acceptable for masonry mortars. Sand No. 18 had an acceptable fineness modulus, but produced a mortar of poor workability and was associated with high percentages of mortar joint cracking.

Effectiveness of the Standard Grading Requirements for Masonry Sands:

It is interesting to examine how the sands studied would have fared if judged by the ASTM standard specifications for aggregate for masonry mortars.[6] Sands Nos. 2, 3, 4, and 6 would not have passed the standard grading requirements, yet they all have excellent results. Sand No. 2 was lacking in fines, and sands Nos. 4 6 had too much in one sieve fraction. Sand No. 3 failed on two counts. It was very deficient in fines and violated the maximum permitted for the fraction between the No. 30 and No. 50 sieves. Yet it was the preferred sand of several masons, made highly workable high-lime mortars, and produced three moisture-proof building units where the cracking between brick and mortar was low. The unsatisfactory sand No. 18 would easily have passed the standard specification requirements, but it was associated with high percentages of mortar joint cracking and produced mortars of poor workability.

Suggested Grading Requirements for Masonry Mortar Sands:

To set up new grading requirements for masonry sands from the sieve analyses and the workability of the mortars produced by 22 sands and from the mortar joint cracking that occurred with 13 is hazardous. But when there is evidence that the accepted requirements may reject excellent sands and pass unsatisfactory ones, a new approach is justified. Tentative grading requirements based upon field performance and a common sense concept of the roller-bearing and void-filling action of the particles of sand in the fractions between successive pairs of sieves as set forth in Table IV are therefore suggested for consideration. These grading requirements have proved to be of value in selecting sands for mortars used in field construction, but need further confirmation.

The suggested requirements for the grading of sands for masonry mortar are drawn exclusively from data on natural pit sands. Some of the percentages are admittedly estimates, but the major requirements are indicated by the data. Natural sands tend to be rounded and probably produce more workability than manufactured sands made by crushing stone or gravel. Whether or not manufactured sands would require the same gradings as pit sands is not known.

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[6] Tentative Specification for Aggregate for Masonry Mortar"(C 144 - 52), 1952 Book of ASTM Standards, Part 3, p. 377.

TABLE IV.—SUGGESTED GRADING REQUIREMENTS FOR MORTAR SANDS.

Fractional Divisions from Sieve Analysis	Minimum Per cent	Maximum Per cent
Retained on No. 4................	0	1.0
Amount between No. 4 and No. 8..	0	6.0
Amount between No. 8 and No. 16.	3.0[a]	20.0[b]
Amount between No. 16 and No. 30.	15.0[c, d]	35.0[e]
Amount between No. 30 and No. 50.	30.0[f]	60.0[g]
Amount between No. 50 and No. 100	5.0[h]	24.0
Amount passing No. 100...........	1.0	10.0[i]

[a] The amount between the No. 8 and No. 16 sieves shall be at least double the amount retained on the No. 8 sieve.

[b] The amount retained on the No. 16 sieve shall not exceed 25 per cent.

[c] The amount retained on the No. 30 sieve shall be not less than 20 per cent.

[d] The amount between the No. 16 and No. 30 sieve shall exceed the amount between the No. 8 and No. 16 sieve by not less than 25 per cent.

[e] The amount retained on the No. 30 sieve shall not exceed 50 per cent.

[f] The amount between the No 30 and No 50 sieves shall exceed the amount between the No. 16 and No. 30 sieves by not less than 15 per cent, and preferably by more than 30 per cent.

[g] The amount between the No. 16 and No. 50 sieves shall not exceed 82 per cent.

[h] The amount between the No. 50 and No. 100 sieves shall exceed the amount passing the No. 100.

[i] The amount passing the No. 50 sieve shall not exceed 25 per cent.

SUMMARY AND CONCLUSIONS

This paper records the sieve analyses of twenty-two sands used in masonry mortar, the workability of the mortars produced by these sands as judged by masons, and the percentages of mortar joint cracking at existing buildings associated with those mortars which were used under conditions favorable to low percentages of cracking. The data are sparse, but it is believed that the following conclusions regarding the sands studied are justified under the conditions of their use in the mortar mixes and at the building units covered in this investigation. The suggested grading requirements for sands for masonry mortar should be regarded as tentative and needing confirmation. The conclusions are as follows:

1. The sieve analyses for all the sands which produced mortars of good or excellent workability had the following characteristics:

   (*a*) The fractional amount between each successive pair of sieves increased from coarse to fine up to and including the amount between the Nos. 30 and 50 sieves.

   (*b*) The fractional amount between the Nos. 30 and 50 sieves exceeded the amount between the Nos. 16 and 30 sieves by not less than 32 per cent.

   (*c*) The fractional amount retained on the No. 16 sieve did not exceed 22 per cent.

   (*d*) The fractional amount retained on the No. 30 sieve did not exceed 47 per cent.

2. A heavy concentration in the amount passing the No. 50 sieve in one sand apparently impaired the workability of the mortar.

3. All sands which were associated with mortar joint cracking under 12 per cent had the sieve analysis characteristics of those sands which produced highly workable mortars, and, in addition, had less than 24 per cent passing the No. 50 sieve.

4. The sands which produced highly workable mortars but had more than 28 per cent passing the No. 50 sieve, were associated with high percentages of mortar joint cracking.

5. The sands which had sieve analysis characteristics different from those given in the first conclusion generally produced mortars with poor workability, but in two instances, a mortar was rated as having fair workability.

6. The average percentage of mortar joint cracking which was associated with the sands producing poor workability mortars or having more than 28 per cent passing the No. 50 sieve was more than

three times as great as the average percentage of cracking associated with those sands producing highly workable mortars and having less than 24 per cent passing the No. 50 sieve.

7. New grading requirements for sands for masonry mortar based primarily upon the percentages between each successive pair of sieves in the sieve analysis are given. They apparently constitute an improved method of judging the suitability of sands.

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DISCUSSION

MR. F. O. ANDEREGG[1] (*presented in written form*).—About 15 years ago Mr. Connor showed me what he meant by "shrinkage cracking." Wherever the mason had failed to push the mortar up against the brick so that a tiny valley was left, Mr. Connor called this separation "cracking," marked *C* in the accompanying Fig. 1.

More recently, Mr. Connor called such an occurrence, which is obviously the result of the mason's technique, "separation cracking." This has probably been most of the 10 per cent initial cracking he speaks about. However, he has come back to the construction project after a few years and made his measurements over again and reported to me something like 40 per cent cracking. He has shown me two instances where the increase is apparently due to the development of real cracks between bricks and mortars. Figures 2 and 3 are photographs taken on one of Mr. Connor's jobs which show obvious cracking, *X*, between bricks and mortar. For instance, a crack in a head joint will frequently continue across the adjacent mortar in the bed joint, and even across the bricks themselves, *B*.

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[1] Consultant, Somerville, N. J.

These cracks have apparently resulted from a variety of forces, which are obviously of some magnitude and which have placed the interfacial bond layer between the bricks, as well as the bricks themselves, under strains greater than they could take. These forces include (1) differential moisture movement, (2) differential thermal movement, (3) wind sway, (4) vibrations from passing traffic, and (5) possibly some loading within the building. The last is probably of little importance in the examples cited by Mr. Connor.

About 25 years ago I had the opportunity of making a study on the effect of aggregate grading on mortar properties.[2] In Table III of that paper the results obtained with different continuous gradings, with an intermittent grading, and with Ottawa standard sand are given. The last two are gap gradings, more or less like those Mr. Connor prefers and feels have reduced the cracking resulting from differential movements in masonry. These two gap gradings showed the smallest moisture movements. This question of the effect of sand gradings on mortar needs more study.

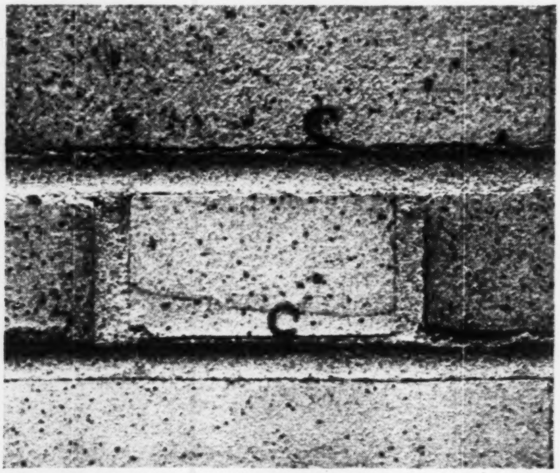

FIG. 1.—Shadow Lines Marked *C* Referred to as Shrinkage Cracks by Mr. Connor and as Technique of the Mason by Others.

(*Courtesy J. M. Hardesty*)

MR. C. C. CONNOR (*author's closure*).—There is no doubt that the separations between brick and mortar increase with time under unfavorable conditions. Joint cracking as high as 10 per cent immediately after the bricks are laid is unusual, but with mortars of poor workability or brick having a high rate of absorption, the cracking may increase to as much as 20 per cent within 48 hr. Such high early cracking continues to grow slowly for a number of years, but if

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[2] F. O. Anderegg, "Grading Aggregates II—The Application of Mathematical Formulas to Mortars," *Ind. Eng. Chem.*, Vol. 23, p. 1061 (1931).

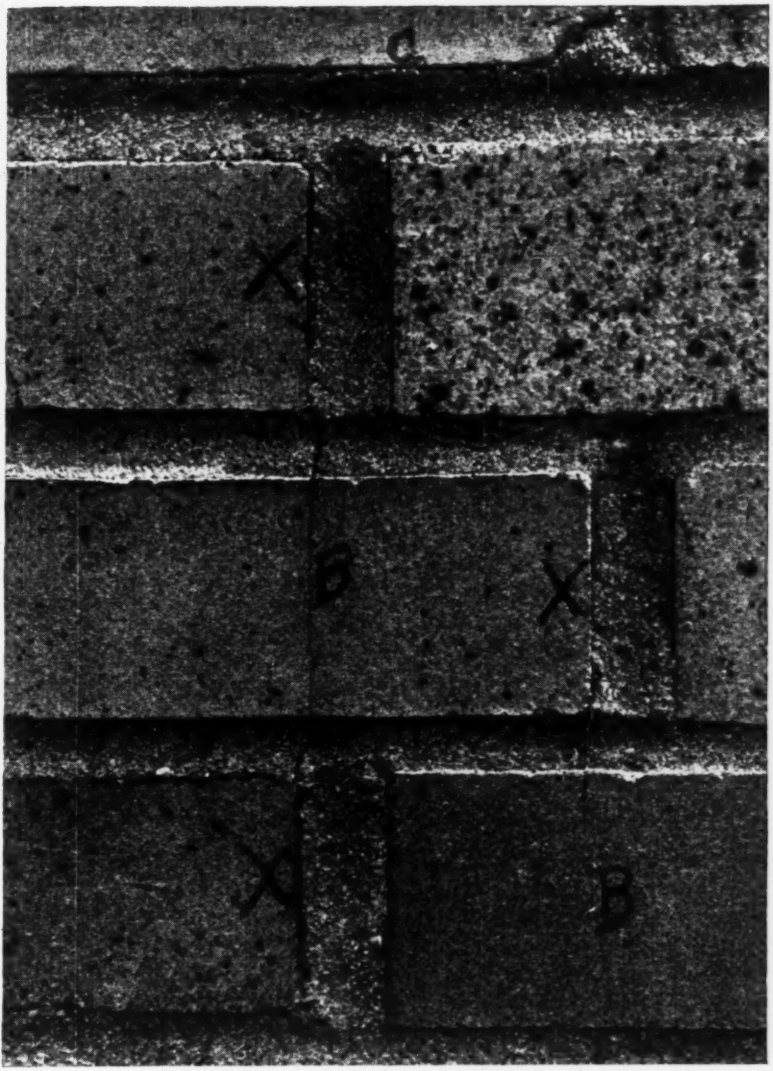

FIG. 2.—Definite Cracks (X) Between Bricks and Mortar Cross Adjacent Mortar Joints and Pass Through Some of the Bricks.

(*Courtesy J. M. Hardesty*)

the early cracking is low there is little or no growth for many years except where the sand contains an excess of material passing the No. 50 sieve.

Mr. Anderegg's statement that he found the smallest moisture movements in mortars containing two sands having gap gradings somewhat similar to those in my paper which were associated with low joint cracking may be quite significant. Mortars containing well-graded sands apparently require less water to produce trowelability satisfactory to the mason than those with poorly graded sands, and a minimum of excess water should reduce the initial shrinkage and the attendant early cracking. Subsequent cracking would be reduced if there were a high extent of bond and a small moisture movement.

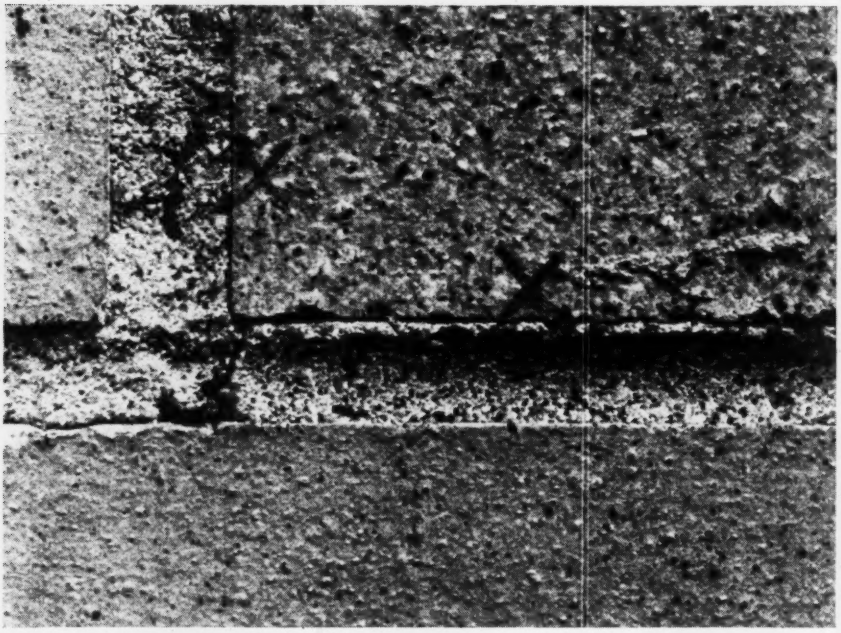

FIG. 3.—More Cracks (X) Between a Brick and Adjoining Mortar.
(*Courtesy J. M. Hardesty*)

Unquestionably the effect of sand grading needs more study. There are indications that its influence on the cracking of concrete is fully as important as on mortar. I believe that further research would be most rewarding.

POROSITY DETERMINATIONS AND THE SIGNIFICANCE OF PORE CHARACTERISTICS OF AGGREGATES*

By D. W. Lewis,[1] W. L. Dolch,[1] and K. B. Woods[1]

Synopsis

This paper has been developed from material obtained from a review of the literature covering research data collected from the laboratory and the field.

The historical aspects of the paper include references in the fields of building stone, brick, and aggregates used in portland cement concrete. Attention is directed toward various methods of test which have been employed for determining the characteristics of pores, and emphasis is given to the relationship of these characteristics to absorption and degree of saturation of aggregate and to the durability of concrete.

It is concluded that the abundance, shape, size, and continuity of pores influence the physical properties and the chemical stability of aggregates and control certain durability characteristics of portland cement concrete. It is concluded further that there is a definite need for research in connection with the development of methods of test for the determination of pore characteristics.

At the end of the paper is a list of selected references.

The major purpose of this presentation is to review pertinent information on the porosity of coarse aggregates used in portland cement concrete. The subject of aggregate porosity is important, especially from the standpoint of concrete durability, and is attracting increasing attention among concrete technologists.

This presentation has been developed from material available in the literature and is divided into two main parts. The first of these deals with methods of measuring porosity and the second with the influence of the porosity of coarse aggregate on the durability of portland cement concrete made therewith.

Measurement of Porosity

The term pores is used interchangeably with voids in this paper and refers to the space in the interior of the individual piece of aggregate unoccupied by solid matter. The term does not refer to the space between pieces of aggregate, which is the "void space" measured in accordance with ASTM Method of Test for Voids in Aggregate for Concrete (C 30 - 37).[2]

Porosity is expressed either as a ratio of the void volume to the bulk volume of the aggregate or as a ratio of the void volume to the solid volume. The first ratio is frequently called the percentage porosity and the second, the voids ratio.

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[2] 1952 Book of ASTM Standards, Part 3, p. 977.

The void system of a solid has been classified in various ways, for example, on a basis of mode of origin, location, or shape of the void spaces. The classification of coarse aggregate porosity of most interest to the concrete technologist is, of course, the one which provides a correlation with the durability of concrete.

The size of pores of greatest importance is difficult to specify. Correlations of porosity of aggregate with durability of the concrete—as measured by freezing and thawing—have been obtained involving 4 to 5 μ as the critical diameter, that is, a relatively large volume of pores which are smaller than this figure in any given coarse aggregate gives rise to a lack of durability of concrete fabricated with the aggregate.

The methods included here for determining porosity cover those used on oil-bearing rocks, cracking catalysts, ceramic materials, hardened portland - cement paste, porous glasses, building stone, and brick as well as those used in studies conducted on coarse aggregates. This departure from confining the discussion to those methods used strictly for aggregates is required to obtain reasonable completeness and implies that some, at least, of these methods can be adapted to give information useful in the study of aggregate porosity. The various techniques have been divided into three classes: (*a*) those which measure the total porosity, (*b*) those which give a simple indication of pore size, and (*c*) those which determine the pore-size frequency distribution.

Methods Which Determine Total Porosity:

The most frequently used method for determining the total porosity of aggregates involves the determination of specific gravities. Definitions of the various specific gravities are given in ASTM Definitions of Terms Relating to Specific Gravity (E 12 - 27).[3] By means of simple calculations the voids ratio can be determined from true and bulk specific gravity or from true and apparent specific gravity and absorption.

True specific gravity is determined pycnometrically. With relatively large samples, the material is powdered and the specific gravity is determined by ASTM Method of Test for Specific Gravity of Soils (D 854 - 52)[4] or some analogous method, using water as the pycnometric fluid. Other liquids can also be used. A volumenometer can be used in which a gas is the displacement medium. The apparatus permits expansion or contraction of a system by a known volume and the measurement of the system's pressure before and after the volume change. By assuming that the gas laws hold, the volume of the system can be calculated with and without the sample in place and a subtraction gives the volume of solids. The apparatus can also be calibrated with standards of known volume. The gas used is generally air or helium, the latter being used if the sample strongly adsorbs air. Simple forms of the apparatus are given by Washburn and Bunting (**46**)[5] and by Karns (**19**). Modifications are given by MacGee (**23**), Nissan, *et al.* (**25**), and Smith and Howard (**40**). Schumb and Rittner (**36**) present a modern apparatus in which helium is employed. Powers and Brownyard (**27**, p. 690) used an apparatus employing an adaptation of this principle. Boehler and Ason (**3**) used the same principle of gas displacement but determined the volume of the system by measuring the time required for the pressure to fall from one value to another while the gas escaped through an orifice.

Apparent specific gravity, bulk specific gravity, and absorption are determined by means of ASTM Method of Test for Specific Gravity and Absorption of Course Aggregate (C 127 – 42).[6] Bulk specific gravity can also be determined by measurement of a symmetrical shape, by coating with paraffin and subsequent weighing immersed in water (applied to compressed bituminous mixtures in Tentative Method of Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 – 51)),[7] or by displacement of mercury. Nissan (24) reviews some of these methods. The bulk specific gravity value that is obtained will depend upon the method used for its determination.

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[3] 1952 Book of ASTM Standards, Part 3, p. 1594.

[4] 1952 Book of ASTM Standards, Part 3, p. 1447.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 957.

Total porosity can also be obtained by use of the so-called McLeod gage type of apparatus devised by Washburn and Bunting (47). In this apparatus a dry sample is immersed in mercury in a closed system. The voids are evacuated by lowering the mercury head. The emergent air is trapped over the mercury and is measured directly.

The disadvantage of the above methods, from the standpoint of the concrete technologist, is that no indication of the size of the pores is obtained.

Methods Which Determine a Simple Size Relationship:

The microscope is a logical tool to use to differentiate between pore sizes. The technique of its use is described by Sweet (42) for stone and by Verbeck (44) for air voids in hardened concrete. The method involves the preparation and microscopic examination of a thin or polished section of the substance. The pore system of a field of this section is duplicated by means of a camera-lucida or photomicrographs and its area is determined with a planimeter and is converted to voids ratio by calculation. By this method the voids larger or smaller than any predetermined size (larger than the limit of resolution of the microscope) can be obtained. Fears (13) made a detailed survey of this method. Its chief disadvantage is that it is time consuming.

One method which gives a definite value of pore size is based on the capillary rise principle. Due to its surface energy, a liquid which wets a solid will be forced into a pore of that solid by a pressure which is inversely proportional to the size of the capillary. An apparatus employing this principle was used by the Department of Scientific and Industrial Research (34) on building stone. An adaptation of their apparatus was reported by Blanks (2). Water was allowed to evaporate from the lower surface of an enclosed cylindrical specimen of aggregate. The pressure drop, due to capillary forces, in a water reservoir on the opposite face was measured as a function of time. The magnitude of the pressure drop varied inversely with capillary size and its speed varied directly with the total number of capillaries. A disadvantage of the method is that the final magnitude of the pressure drop represents the size of the smallest diameter of the largest continuous void traversing the specimen. When a pressure corresponding to this size of capillary is reached, air enters the specimen and the action ceases.

Another method which yields a value of pore size requires a determination of the permeability of a sample to gas or liquid flow. Stull and Johnson (41) used this method on bricks and applied the Poiseuille equation to calculate the average size of pores. The simplifying assumption was made that the pore system was an ideal one of identical, uniformly distributed pores. Most porous substances probably depart radically from such an ideal structure. Schwertz (37) used permeability methods in his study of porous glasses. His air-diffusion apparatus was a simple one wherein the porous material was the barrier to an evacuated bulb and the pressure rise of the system as air flowed through the barrier was measured as a function of time. The application of Knudsen's equation gives the average size of pores. Schwertz also measured permeability to liquids and used Poiseuille's equation to calculate pore size. Structure constants were used in these calculations to account for the departure from ideality of the pore system.

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[6] 1952 Book of ASTM Standards, Part 3, p. 967.
[7] 1952 Book of ASTM Standards, Part 3, p. 1368.

For extremely small pores the variation in accessibility to various sized molecules can be measured (see review by Emmett (**12**)). For pores in a larger size range the variation in absorption of liquids of different penetrating abilities (**33**) will give a rough estimate of pore size.

Methods Which Determine the Pore-Size Frequency Distribution:

If V, the volume of pores which are smaller than a certain radius r, is obtained as a nonlinear function of r, then dv/dr is also a function of r—the distribution function for pore size. A plot of dv/dr *versus* r is the pore-size frequency distribution curve and shows the relative volume contributed by pores of any certain size. Such a curve for coarse aggregates would be extremely desirable for use by concrete technologists.

The subject of adsorption phenomena is one on which a large amount of work has been done. Various theories have been advanced to explain the adsorption process. An adequate explanation of these theories cannot be given briefly. The reader is referred to Braunauer's book (**4**) and to reviews by Emmett (**12**) and Foster (**14**). The data from adsorption measurements are generally presented in the form of an isotherm, that is, a plot of the amount of adsorption at constant temperature as a function of the pressure of the system. These data can be used to calculate the surface area of the adsorbent. Knowing total pore volume and surface area, the hydraulic radius of the pores can then be calculated. The distribution curve can be determined using certain other theories (**14, 39**), but, in general, only for pores smaller than several hundred Ångström units in diameter. A large amount of experimentation has been done with a great many different absorbents and adsorbates. Of particular note to concrete technology is the work by Powers and Brownyard (**27**) on adsorption of water vapor by hardened portland cement paste. Recent work has been done on determining the specific surfaces of coarse aggregates by means of low-temperature nitrogen adsorption (**1**).

One of the most promising methods for the determination of the pore-size frequency is that originated by Ritter and Drake (**29**) using a mercury porosimeter. The principle involved is that, due to its surface energy, a liquid which does not wet a solid is denied entrance to a pore of that solid by a pressure which varies inversely with the size of the pore.

In the mercury porosimeter the sample is evacuated and immersed in mercury in a dilatometer. The capillary pressure is then opposed by external pressure. The change in volume is measured as the pressure is increased and the mercury fills successively smaller voids. The distribution curve is readily obtained from the data. Ritter and Drake (**9, 10, 29**) used their apparatus on a variety of substances and operated at pressures up to 60,000 psi to cover the range of very small pores. Purcell (**28**) adapted this method to cover the size range of 0.1 μ diameter and larger pores (0 to 2000 psi) for use on oil-bearing rocks.

Another method which could give a distribution curve is the capillary diaphragm method (**16**) used by petroleum technologists for the determination of capillary pressures in rocks. In this method the saturated sample is connected through a finely porous, saturated diaphragm and a calibrated tube, to a vacuum pump and manometer. The capillary pressure is opposed by atmospheric pressure as the vacuum is applied and water will leave the sample. The loss of water is measured in the calibrated tube and an experimental plot is obtained of capillary pressure (a function of void size) *versus* degree of saturation (a function of void volume). The disadvantage is the limit imposed by the entrance of air into the diaphragm.

To overcome this difficulty, Hassler and Brunner (**16**) developed a centrifugal method. The saturated sample is placed in a special centrifuge and is whirled at increasing speeds. The centrifugal force opposes the capillary force tending to hold the water in the voids. The water lost is measured in a calibrated tube attached to the sample and observed in motion by means of stroboscopic light. Again, a plot of degree of saturation *versus* capillary pressure is obtained.

A final method to be mentioned is the small-angle X-ray scattering technique of Ritter and Erich (**30**). These authors applied the method to a pore-size range of approximately 0 to 400 Å. The necessity for special equipment is obvious.

SIGNIFICANCE OF POROSITY OF CONCRETE AGGREGATES

The characteristics of the internal pore space in concrete aggregates have been described as the most important of all aggregate physical properties (**2, 32**). The importance of porosity is due to its influence on the other physical and chemical properties of the aggregate and to its control of durability in freezing and thawing. The abundance, size, shape, and continuity of the pores determine the amount of water the aggregate can absorb, its absorption rate, its ease of draining (or the water-retention properties), its internal surface area, and the portion of its bulk volume that is occupied by solid matter.

Effect on Chemical and Physical Properties:

Aggregates having large total pore volumes, especially if the individual pores are relatively small in size, have greater specific surfaces that may be susceptible to chemical attack than do those with smaller pore volumes or larger sizes of pores. Thus, for an aggregate type that is subject to chemical reactions in concrete, the rate of reaction and the chemical stability are influenced by the porosity characteristics.

The physical properties of bulk specific gravity, strength, elasticity and abrasion resistance are similarly affected by the porosity (**32**). Larger void volumes result in smaller proportions of solids per unit bulk volume. In addition to the obvious effect of reduced specific gravity, the more porous materials have a smaller net area of solids to resist any load to which the particles are subjected. For aggregates of similar composition, therefore, the more porous materials will have the lower strength values.

The bond of cement paste to the aggregate particles is determined by complex and poorly understood physicochemical properties of the surface and by the surface texture. Both roughness and the pore characteristics of the surface zone affect the surface texture and the quality of bond with the cement paste. Pore characteristics that permit no penetration of the surface of the aggregate particle are not conducive to a good bond (**31**).

Aggregate thermal characteristics are influenced by the porosity. In an indirect manner, the porosity affects the thermal properties through control of water absorption. Reports by Lu (**22**), Fox and

Dolch (15), and Clark (6) indicate significant changes in coefficient of expansion, diffusivity, and conductivity, respectively, with change in the moisture content of the stone. It is probable that the relative proportions of pore space and solids also have an influence on the thermal properties of dry aggregates.

Relationship of Porosity to Durability of Concrete in Freezing and Thawing:

The most significant effect of porosity characteristics on the suitability of concrete aggregates is the effect on the durability of the concrete in freezing and thawing (2, 32). Materials that contain no water are not likely to be harmed by freezing; for damage to occur, critical conditions of water content and lack of drainage must be present. The amount of water that an aggregate can absorb is controlled by both the total porosity and the size of the pores. Neither very large pores nor pores of capillary size, connected to the surface by smaller pores or surrounded by mortar having a smaller pore size, will fill with water by capillarity. Pores of smaller sizes are subject to greater capillary pressures and may fill readily when a source of water is available. The size and continuity of the pores control the rate of absorption and, similarly, the rate at which the water can escape from the aggregate particle. To obtain complete information on the porosity of an aggregate, measurement of the characteristics of total porosity, size frequency distribution, shape, and continuity would be required.

Of these characteristics, only the total porosity has been measured adequately by test methods actually applied to concrete aggregates. As noted under Measurement of Porosity, several methods for determining other pore characteristics exist but have not been applied to aggregates. The lack of extensive porosity test data on aggregates has precluded any general correlation of durability with actual porosity characteristics. Instead, past investigators have attempted to make correlations of durability with either a single porosity measurement or some physical characteristic that is dependent upon porosity. Included in these categories are correlations of durability with the ratio of volume of voids smaller than 5 μ in diameter to the volume of solids and with the physical characteristics of absorption, specific gravity, and degree of saturation. The correlations obtained are, in general, limited in usefulness to the materials tested and to similar materials exposed to the same conditions. Despite the lack of any general correlation of freezing-and-thawing durability with the fundamental porosity characteristics, the studies that have been made serve to emphasize the importance of porosity and have led to the development of definite theories of the mechanism of disruption.

Correlations.—Investigations of the porosity of concrete aggregates have been conducted almost entirely during the last 20 years. Prior studies of porosity were made on building stone and brick; the techniques developed were later applied to aggregates.

Attempts to correlate the durability of building stone with the absorption and specific gravity date back to at least 1847 (7). Later work by Hirschwald (17) and Howe (18) utilized total porosity measurements and calculation of the degree of saturation. Howe (18) noted that the type of porosity was more important than the total pore volume and that the degree to which the pores became filled with water under natural conditions determined the durability in freezing and thawing. Similar porosity studies on brick and ceramics were made by Douty and Beebe (11), Washburn (45) and others.

Seipp (38) calculated that, theoretically, the degree of saturation of building stone should be less than about 0.91 to obtain good durability. Higher degrees of saturation would leave insufficient space for the expansion that occurs when the water freezes. Kreuger (20) later found critical degrees of saturation on the order of 0.85, due apparently to nonuniform distribution of water in the pore system. Some of the pores could be almost completely saturated while others remained relatively empty, but extensive damage from freezing and thawing would still result.

Schaffer (34) reported test results of pore-size determinations on building stone. The volume of pores having diameters less than 5 μ was correlated with the resistance of the stone to freezing and thawing.

The earliest correlations of the porosity properties of concrete aggregates with durability appear to be confined to cherts. Cantrill and Campbell (5), Wuerpel and Rexford (49), and Sweet and Woods (43) correlated the freezing-and-thawing durability of chert with such factors as absorption, bulk specific gravity, pore structure, and dye penetration. These correlations, although imperfect and limited to a specific material, are important in establishing the influence of porosity on the durability of concrete aggregates.

Wray and Lichtefeld (48) reported the results of freezing-and-thawing tests on gravels with varying degrees of saturation. The more water the aggregate contained, the poorer was the durability. Later work reported by Sweet (42) correlated degree of saturation with both laboratory freezing-and-thawing tests and the field durability of concrete in which the materials were used.

Correlation of the volume of pores less than 5 μ in diameter with freezing-and-thawing durability of Indiana limestones has been reported by Sweet (42) and Fears (13). The aggregates with the poorest durability had voids ratios of the smaller pores several times as great as did some of the most durable materials. Blanks (2) reported a similar correlation and stated that the pores having a diameter less than 4 μ were especially critical.

The application of porosity studies to concrete aggregates has paralleled, in both techniques and results, the previous work on building stone. The majority of the correlations established were of durability with some property affected or determined by the porosity characteristics rather than with the fundamental porosity characteristics themselves. As a result, serious limitations are placed on the applicability of the correlations to concrete aggregates in general. Despite the limitations, such correlations are superior to those of standard tests in many cases and have been suggested by some investigators for acceptance testing of aggregates (5, 43, 21).

Mechanism.—The durability, in freezing and thawing, of an aggregate is primarily dependent upon its ability to attain—and retain—a high degree of saturation under the given conditions of exposure. The harmful pore size is large enough to permit water readily to enter a high percentage of the pore space but not so large as to permit easy drainage. According to Dorsey (8), the force exerted by freezing water (if no expansion or escape of water is possible) ranges from 0 psi at 32 F to about 29,000 psi at −4 F. Pressures in this range may be developed in aggregate particles when the degree of saturation is so high that the remaining void space is less than the expansion that takes place when the pore water freezes. To avoid the development of pressures in excess of the tensile strength of either the aggregate particle or the surrounding mortar, the pore water must be able to flow into unfilled pores in the stone or escape from the aggregate particle. Escape from the aggregate particle may be blocked by a frozen zone around the outside, resulting in the development of high static pressures in the interior. Even when flow away from the freezing zone is possible, the hydraulic pressures necessary to cause movement through small capillaries may be so high as to cause disruption of the material. Blanks (2) states that pores less than $4\,\mu$ in diameter will drain effectively only at pressures high enough to cause failure of some rocks and concretes in tension. It is not necessary that the aggregate particle itself be disrupted for the concrete in which it is used to suffer damage from freezing and thawing (35). The aggregate may have sufficient strength and elasticity to withstand the imposed stresses without failure, but with disruption of the surrounding mortar by the expansion of the particle. Freezing-and-thawing tests of the aggregate alone may not, therefore, be indicative of its effect in concrete (35, 42). The rate at which water must escape from the freezing zone and the pressures required to cause such flow are dependent upon the rate of freezing and the permeability (pore size and continuity) of the aggregate. An hypothesis of this nature for the effect of freezing on concrete has been presented and discussed in detail by Powers (26).

The most important aggregate properties that control the freezing-and-thawing durability, according to the hypothesis discussed above, are the pore-size distribution and the permeability of the material. Since the permeability is controlled by the size and continuity of the pores, these two porosity characteristics may be considered as the important ones, with total porosity of secondary importance. No satisfactory tests to evaluate these characteristics have yet been applied to concrete aggregates, and the need for further investigation is apparent.

SUMMARY

With a property as important as pore space, it is surprising that more emphasis has not been given to this subject—not only by research workers in the fields of aggregate and concrete technology but also by those interested in specifications and in the development of methods of tests. It would be difficult to prove that any other physical property is of greater importance than the porosity characteristics (amount, size, and continuity of the pores) in either natural or artificial aggregates. The pore characteristics of a coarse aggregate not only influence the physical durability properties of concrete but also any potential chemical reaction. It is apparent from the information collected in this paper that research in this area should be extremely fruitful. Immediate attention should be given to methods of test, particularly with respect to methods used in related fields for determining pore characteristics of porous materials. It is probable that specification writers will be seeking material of this character in an attempt to obtain the best quality of aggregate for use in various types of construction.

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DISCUSSION

MR. J. M. RICE[1] (*presented in written form*).—The authors of this paper are to be highly commended for focusing attention on pore characteristics and particularly for summarizing the test methods that have been utilized by other industries. We are among those who have neglected this important phase of aggregate technology.

However, for the past five years, we have been testing ledge rock by a method which measures the harmful effect of internal frost action or ice formation. The most obvious result of internal ice formation is the expansion or cracking of the aggregate. To measure this effect $1\frac{3}{4}$ by 2-in. cores are drilled from ledge rock, the ends are sawed and ground, and then the linear expansion caused by freezing and thawing is measured by means of the comparator shown in Fig. 1.

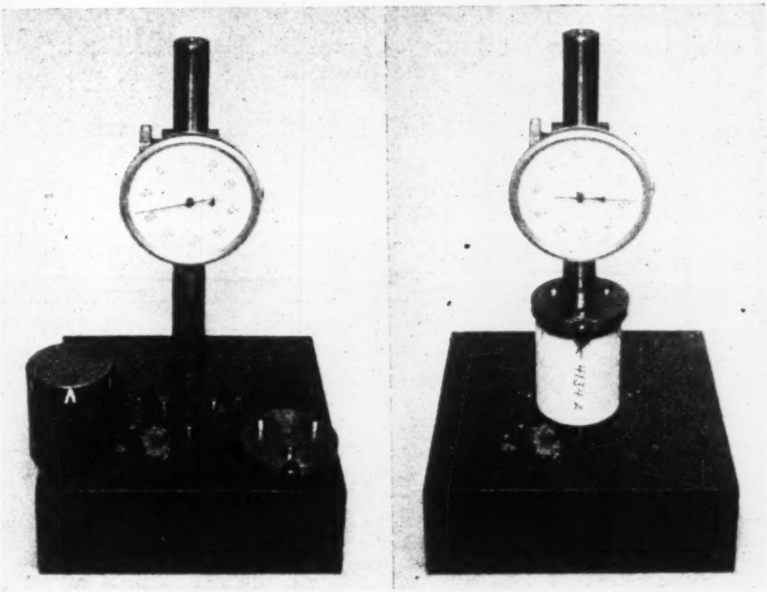

FIG. 1.—Core Length Comparator.

While developing this test method, we experimented with the initial saturation condition of the cores, that is, either by vacuum or by 24-hr immersion. Currently we believe that the most realistic saturation treatment is that which approximates the conditions under which the aggregate is actually to be used.

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[1] Research and Testing Engineer, National Crushed Stone Assn., Washington, D. C.

Unless a stone is to be used in a quarry-wet condition without preliminary drying in stockpile, the cores are presaturated by 24-hr immersion from an air-dry condition. The cores are frozen in air, not water, but drying is retarded by wrapping the cores with aluminum foil. After overnight freezing, the cores are thawed in air for 4 hr and then reimmersed in water for 4 hr.

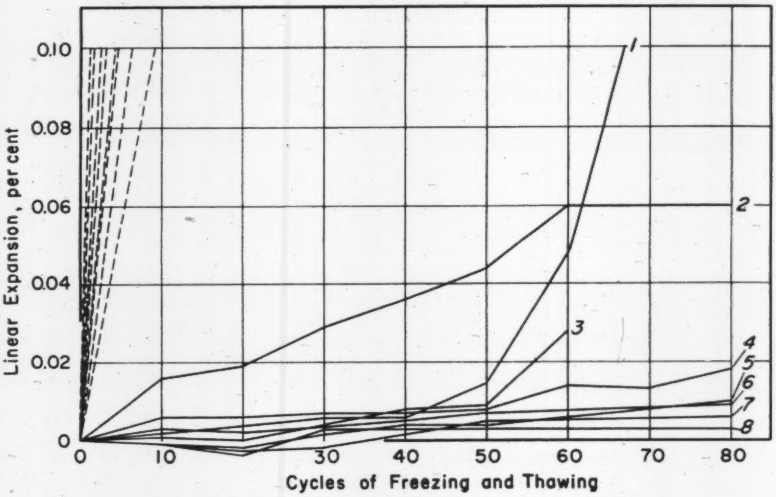

FIG. 2.—Effect of Initial Saturation on Expansive Ledges.

Ledge	– – – Cores Vacuum Saturated	—— Cores Immersed 24 hr		
	Vacuum Absorption, per cent	Saturation, per cent		
		Vacuum	24 hr	80 Cycles
No. 1	3.42	99.8	63.9	...
No. 2	3.24	94.7	71.1	95.6
No. 3	3.52	100.0	60.7	...
No. 4	3.94	100.0	62.8	85.2
No. 5	6.01	100.0	56.9	80.5
No. 6	2.76	99.6	45.0	80.6
No. 7	5.08	100.0	51.1	85.3
No. 8	2.62	97.5	50.9	70.9

Figure 2 shows the effect of the initial saturation conditions on the expansion of eight ledges which are characterized by excessive expansion *when highly saturated.* These ledges are from three Midwest limestone quarries which have been reported to be sources of aggregates with poor performance records. All of the vacuum-saturated cores showed high degrees of saturation and very rapid expansion—more than 0.1 per cent in ten cycles. Similar cores, when only partially saturated by 24-hr immersion performed much better. Two of the ledges (Nos. 1 and 3) eventually failed, but the remaining six were sound after 80 cycles even though they became more highly saturated.

Pore studies of the above ledges would probably indicate potential unsoundness, yet the actual performance of the aggregates in concrete would be determined by the initial and subsequent saturation conditions. It is possible that critical degrees of saturation would never be attained if the concrete mortar exerts a preferential absorption for the available water.

The above results would tend to confirm the observation that highly saturated, absorptive aggregates are not durable. However, there are notable exceptions such as are illustrated in Fig. 3. These ledges represent two Midwest limestone quarries that have good service records. All of these cores had relatively high degrees of saturation and high absorption, yet they performed well in the expansion test.

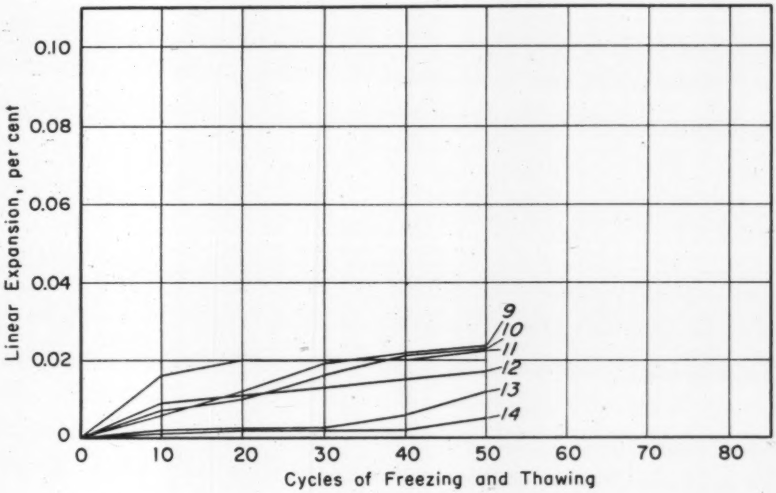

FIG. 3.—Absorptive Ledges with Low Expansion. Cores Vacuum Saturated

Ledge	Vacuum Absorption, per cent	Saturation, per cent	
		Vacuum	50 Cycles
No. 9	3.29	82.9	84.5
No. 10	3.49	83.0	78.1
No. 11	8.40	94.2	92.8
No. 12	7.07	100.0	99.2
No. 13	2.69	74.9	78.6
No. 14	4.60	83.9	76.9

Where good aggregates are abundant, a consumer might be justified in adopting criteria, such as a maximum percentage absorption and degree of saturation, that would only occasionally exclude sound aggregates. Where aggregates are scarce, specifications should not be so restrictive. On the other hand, the general adoption of such criteria may admit deleterious aggregates. Figure 4 shows the expansion curves for several cores with low water absorption. These cores represent ledges from two limestone quarries in the East which may be identified as argillaceous and carbonaceous. Complete pore studies of these materials would certainly be of interest, although it is possible that low tensile strength or even expansive minerals may have been factors contributing to failure.

It is our opinion that a complete

knowledge of pore characteristics should be helpful in ascertaining whether or not a particular aggregate is susceptible to failure due to internal frost action. With this information and some knowledge of the strength characteristics of the aggregate and the saturation conditions to be encountered, the performance of the aggregate may be predictable.

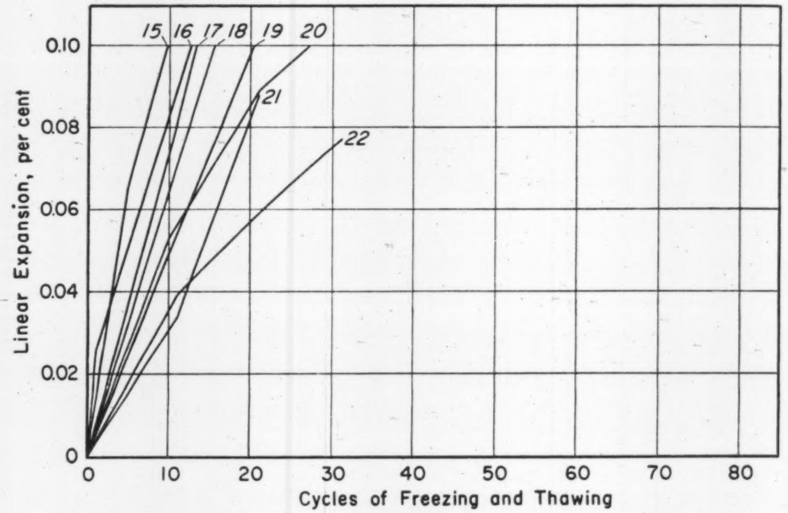

FIG. 4.—Nonabsorptive Ledges with High Expansion.

CORES IMMERSED 24 HOURS

Ledge	Absorption, per cent	
	Vacuum	24 hr
No. 15	0.40	0.27
No. 16	0.87	0.66
No. 17	1.77	1.69
No. 18	1.06	0.53
No. 19	1.82	1.13
No. 20	0.85	0.59
No. 21	1.31	0.87
No. 22	0.86	0.64

MESSRS. D. W. LEWIS, W. L. DOLCH, AND K. B. WOODS (*authors' closure*).—Mr. Rice's data are interesting and pertinent to the problem at hand. They show once again that no single property, such as absorption or degree of saturation can unreservedly be depended on for performance prediction. They reinforce the authors' contention of the need of a more fundamental description of the pore system of aggregate materials. Adequate test methods are needed. Perhaps when they have been devised and applied a consistent picture of aggregate performance, as influenced by porosity, can be set forth.

THERMAL EXPANSION TESTS ON AGGREGATES, NEAT CEMENTS, AND CONCRETES*

By Leonard J. Mitchell[1]

Several investigators during the past decade have shown that low durability of concrete could be caused by thermal incompatibility between the cement mortar and the aggregate. This incompatibility has generally been attributed to wide differences in the coefficients of thermal expansion of the various materials. The Bureau of Reclamation laboratories have for many years investigated the effects of thermal characteristics of aggregates on the durability and other properties of concrete. The present program was initiated in 1948 to study the incompatibility problem and provide data for general use in concrete design and control. Special equipment was designed to measure accurately length changes in small test specimens.

Tests were made on 41 aggregate minerals, 11 portland cements, 2 portland-pozzolan cements, 14 mortar mixes, and many plain and air-entrained concretes. Other variables investigated were moisture content, mix proportions, air content, and test methods.

Aggregates have widely varying coefficients of thermal expansion and these coefficients are reflected in the values obtained on concrete specimens. The average value of 5.5 millionths per deg Fahr, normally used in design, does not apply for all concretes. Limestones, which may have coefficients as low as 2 millionths per deg Fahr, produce concretes with coefficients as low as 3.2 millionths per deg Fahr. The coefficient of thermal expansion of neat cement ranges from 6 to 12 millionths per deg Fahr, depending primarily on the moisture content. Many other factors cause variations in the coefficients, but most of these differences appear to be directly related to the moisture contents of the specimens. The coefficient of thermal expansion of concrete may be computed, if the correct coefficients of the separate materials are known.

At intermediate degrees of saturation, the coefficient of neat cement paste is greater than at dry or saturated conditions and therefore would increase the potential stresses attributable to thermal incompatibility. When the saturation is above 91 per cent (critical saturation), the disruptive stresses caused by ice pressure are much greater than stresses caused by differences in thermal coefficients. Concretes which absorb moisture readily and are above critical saturation, will fail after only a few cycles of freezing and thawing.

Description of Apparatus

The apparatus used for obtaining thermal expansion of small specimens, Fig. 1, consists of fulcrum-type extensometer frames, electromagnetic strain gages, electronic indicators, appurtenant equipment, and tanks, through which an ethylene glycol solution is circulated at temperatures controlled by refrigeration coils and electric heaters.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Materials Engineer, Bureau of Reclamation Denver, Colo.

Fulcrum-Type Extensometer Frames:

Figure 2 shows that the extensometer frames amplify the movement by a 2 to 1 ratio and may be adjusted to test specimens from 1 to 3 in. in size. The frames are designed so that all significant members are of Invar steel and the most critical members are located in zones of constant temperatures. Thermal effects on the frames are eliminated by the frame design, by calibration, and by test technique. The contact screws and fulcrum rods are in air at room temperature and shielded against radiation from the variable temperature control bath. Only the ends of the main bars and the contact points are immersed. The contact points are very short and the main bars are perpendicular to the movement. Insulating washers and small contacts through bearings minimize conduction to the fulcrum connecting rods. The H-section shape of the main members supplies stiffness with light weight and provides large heat transfer surfaces.

Electromagnetic Strain Gage:

The electromagnetic strain gage is designed to measure movement as small as 3 millionths of an inch. This is ac-

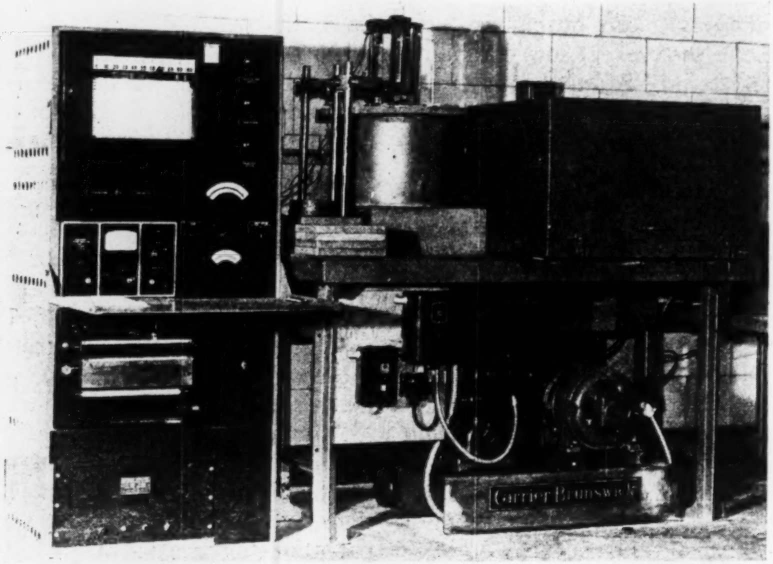

FIG. 1.—General View of Equipment for Measuring Thermal Expansion.

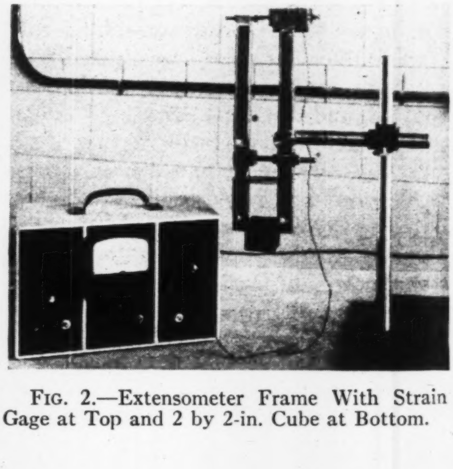

FIG. 2.—Extensometer Frame With Strain Gage at Top and 2 by 2-in. Cube at Bottom.

complished by measuring electrical reactance in an electromagnetic system consisting of a laminated silicon steel armature mounted between two sets of coils on laminated silicon steel cores. Movement of the armature which is attached rigidly to the gage probe causes changes in relative inductance. The gages, when connected to the electronic indicator, form part of an alternating-current bridge, having an output signal amost proportional to the displacement of the armature over a wide range.

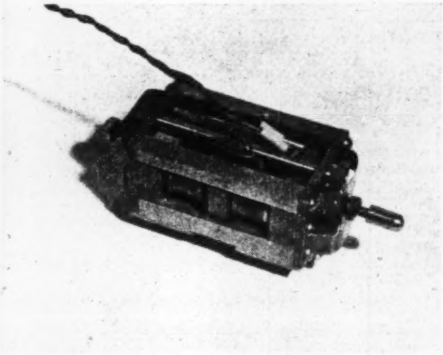

FIG. 3.—Electro Magnetic Strain Gage.

The strain gage, which uses Invar steel in all critical places and waterproof varnish on the wires, is nearly insensitive to temperature and humidity changes. Parts have a minimum of machining and no critical tolerances or fine finishes. Simplicity of design contributes to ruggedness (Fig. 3).

Electronic Indicator:

The electronic indicator consists of a single-frequency audio oscillator, a balancing unit, an amplifier, a phase-sensitive detector or discriminator, and a direct-current microammeter (Fig. 4). A zero adjustment in the indicator allows balancing of the circuit elements to give any desired reference reading on the meter.

The balancing unit provides a method of making both resistive and reactive adjustments to the alternating-current bridge circuit containing the strain gage. The reactive or capacitive adjustment allows compensation for difference between gages. The resistive adjustment balances the bridge output signal from active gages. The change in resistive reading is so nearly proportional to the armature movement of the gages that a straight line can be used for the range of these tests. Five range steps each reading 1000 units supply ample range.

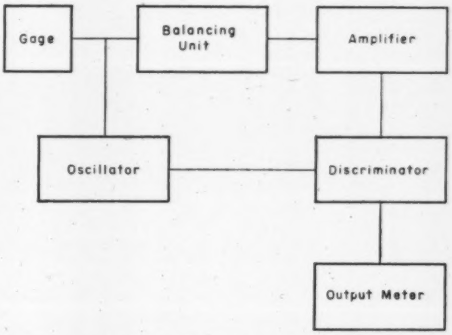

FIG. 4.—Block Diagram of Gage and Indicating Unit.

A gain adjustment in the amplifier controls the sensitivity of the indicator to suit the particular application. For thermal expansion tests, this adjustment is such that one reading unit for a 2-in. specimen is about 1 millionth in. per in.

The indicator was designed to minimize temperature and humidity effects. The resulting stability produces highly uniform and dependable readings.

Temperature Controls:

Temperature control is necessary to determine thermal coefficient of expansion. Two insulated tanks contain the ethylene glycol solution in which the temperature is controlled by means of electric heaters and refrigeration coils.

TABLE I.—THERMAL COEFFICIENT OF ROCK MINERALS FROM VARIOUS SOURCES.

Specimen	Classification—Description and Source	Thermal coefficient, millionths per deg Fahr
	LIMESTONE	
No. HH104	Limestone pebble from aggregate, Hungry Horse Dam, Mont.	3.7
No. M 141	Limestone from tunnel at Hungry Horse Dam, Mont.	4.5
No. M 299-3	Quarried limestone (Cedar Bluff) from Fort Riley, Kan.	3.0
No. M 305-c	Limestone from Pike View, Colo.	2.4
No. M 305-d	Limestone from Pike View, Colo.	3.8
No. M 305-f	Limestone from Pike View, Colo.	1.2
No. M 391-A	Quarried "Cottonwood limestone," Manhattan, Kan.	2.8
No. M 391-B	Quarried "Cottonwood limestone," Manhattan, Kan.	2.6
No. M 497	Quarried "Cottonwood limestone," Manhattan, Kan.	2.2
No. RR104	Pebble from Republican River gravel, Colo.	4.2
No. RR109	Specimens RR109, RR110, and RR111 are three pebbles of chalky, opaline, argillaceous limestone from Republican River gravel	1.9
No. RR110		1.9
No. RR111		1.4
No. P 7736	Quarried limestone from Angostura Dam, S. D.	2.0
No. SML 1	Small slivery samples of crushed siliceous magnesium limestone from California	6.5
No. SML 2		5.2
No. SML 3		5.2
No. p-6	Sandy limestone pebble from gravel, Palisades Dam, Idaho	5.3
No. M 696	Kaibab limestone from near Glen Canyon, Ariz.	4.5
	SANDSTONE	
No. HH102	Meta sandstone from aggregate, Hungry Horse Dam, Mont.	5.8
No. HH103	Sandstone from aggregate, Hungry Horse Dam, Mont.	5.3
No. M 437-1	Hard quartzose sandstone from Coal Creek, Golden, Colo.	6.5
No. M 616	Similar to M 437-1 from quarry at Carter Lake, Colo.	6.5
No. M 617	Similar to M 616 except more chert, Carter Lake, Colo.	5.9
No. M 618	Similar to M 617 except better and with bedding separation	5.9
No. M 619	Similar to the above except poorly interlocked and lightly cemented from quarry at Carter Lake, Colo.	5.7
No. M 442	From sandstone ledge near Moorhead Dam, Mont.	3.7
No. M 520	Calcareous sandstone from near Moorhead Dam	5.7
No. M 453	Opaline sandstone from near Akron, Colo.	5.4
No. RR101	Opaline sandstone pebble from Republican River, Colo.	4.8
No. RR105	Pebble from the Republican River, Colo.	3.4
No. p-4	Fine-grained sandstone pebble from Palisades Dam, Idaho	6.0
	SILTSTONE	
No. p-5	Red siltstone pebble from gravel, Palisades Dam, Idaho	4.1
	ARGILLITE	
No. HH100	Pebble from gravel at Hungry Horse Dam, Mont.	5.4
	QUARTZITE	
No. CC104	Pebble from gravel at Cherry Creek Dam, Colo.	4.8
No. p-1	Pink quartzite pebble from gravel at Palisades Dam, Idaho	5.6
No. p-2	Gray quartzite pebble from gravel at Palisades Dam, Idaho	5.2
No. p-3	Black quartzite pebble from gravel at Palisades Dam, Idaho	5.0
No. M-500	Sample from Wolf Creek siding of the Union Pacific Railroad	4.5
	CHERT	
	Sample from Pickwick Dam, TVA	6.3
	CHALCEDONY	
No. RR103	Pebble from Republican River gravel, Colo.	6.0
	QUARTZ	
No. RR108	Pebble from Republican River gravel, Colo.	8.0
	Mined samples from Buffalo Park, Colo. x_1 faces	5.6
	x_2 faces	6.8
	x_3 faces	7.5
	BASALT	
No. HH107	Pebble from gravel at Hungry Horse Dam, Mont.	4.1
No. M 268	Quarried "Table Mountain basalt," Golden, Colo.	4.0
No. RR102	Pebble from Republican River gravel, Colo.	3.4

TABLE I.—*Concluded*

Specimen	Classification—Description and Source	Thermal coefficient, millionths per deg Fahr
	RHYOLITE	
No. M 281-2	Quarried Crooked River Project, Ore.	4.6
No. RR106	Pebble from Republican River gravel, Colo.	2.3
	RHYOLITIC TUFF	
No. M 431	Light-gray fine-grained sample from Sundance Mountain, Wyo.	4.2
	META ANDESITE	
No. HH106	Pebble from gravel at Hungry Horse Dam, Mont.	3.8
	ANDESITE	
No. RR107	Pebble from Republican River gravel, Colo.	4.5
	PHONOLITE PORPHYRY	
No. M 435	Sample from Barlow Canyon (Keyhole Dam), Wyo.	3.4
	GABBRO	
No. HH101	Pebble from gravel at Hungry Horse Dam, Mont.	4.1
	DIORITE	
No. HH105	Pebble from gravel at Hungry Horse Dam, Mont.	4.3
	GRANITE	
No. CC100	Pebble from gravel at Cherry Creek Dam, Colo.	3.6
No. RR100	Pebble from Republican River gravel, Colo.	4.5
No. M 283	Crushed granite breccia from Davis Dam, Ariz.	2.0
	GNEISS	
No. CC101	Pebble from gravel at Cherry Creek Dam, Colo.	3.6
	SCHIST	
No. CC102	Pebble from gravel at Cherry Creek Dam, Colo.	4.7
No. CC103	Pebble from gravel at Cherry Creek Dam, Colo.	4.0
	FELDSPAR	
	Massive pink sample—between original cleavage faces	9.7
	Massive pink sample—between 0.001 faces	0.5
	Massive pink sample—between 0.010 faces	1.1
	MARBLE	
No. M 439	Georgia commercial marble parallel to the banding	−0.9
No. M 439L	Georgia commercial marble perpendicular to the banding	2.5

The heating coils are turned on and off manually at various intensities, while the refrigeration is controlled automatically at preset temperatures by a resistance thermometer in a mechanical bridge circuit. After the fluid in the main tank reaches control temperature, the fluid in the specimen tank gradually approaches the desired temperature. The circulating pump is stopped when the desired temperature is reached and steady conditions are soon obtained.

Calibrations:

The frames, gages, and indicator are calibrated by reading length changes at various temperatures on metal specimens with known coefficients of expansion.

Frequent check calibrations, which show the setup to be very stable, are reproducible within ±3 per cent. The thermocouple is calibrated by attaching it to the bulb of a precision thermometer and reading both while they are immersed in water in a thermos bottle.

SPECIMEN PREPARATION

Specimen preparation is very simple. Specimens are from 1 to 3 in. long with the ends approximately parallel. No special preparation of the test faces is necessary as troweled, formed, rough sawed, or ground surfaces of either concrete or rock are acceptable. All specimens are measured for length to an accuracy of 1 per cent. The thermal expansion specimens are generally tested in as-received, oven-dried, or vacuum-saturated conditions and are carefully coated with a wax compound to prevent moisture change while immersed in the controlled temperature bath.

TEST PROCEDURE

The fulcrum-type extensometer frame is adjusted to the approximate length of the specimen by moving the jam nuts on the fulcrum bar. A spring is selected which, when fitted in one of the three spring positions, will supply enough tension to hold the specimen between the test points. The specimen is then inserted between the blunt test points at the lower end of the main members of the extensometer frame. The points are placed approximately perpendicular to the surface of the specimen to minimize slipping and are *firmly* pressed through the wax coating. Final adjustment of the jam nuts is made so that the main members of the extensometer frame are within $\frac{1}{32}$ in. of parallel. The gage is next clamped in position with its probe touching the head of the contact screw. The contact screw is adjusted so that gage readings will cover the range of the test. Upon completion of all adjustments, the soundness of the setup is tested by observing the gage reading while the extensometer frame is lightly tapped. If the specimen is properly placed, the gage reading will change only slightly and then remain constant.

Readings are taken by balancing the indicator unit for the approximate capacity characteristic of a given gage, then adjusting the resistive balance and repeating until complete balance is obtained.

DISCUSSION OF TEST DATA

Aggregate:

The length changes of more than 60 rocks representing 21 different mineral aggregates were determined during a temperature cycle of 70 to 15 F and return. A straight-line length-temperature relationship in this temperature range was generally obtained with stable and reproducible results. The sources and thermal coefficients of expansion of these rocks are given in Table I. These data and those obtained by other investigators (**1**, **2**, **3**, **4**)[2] show that the thermal expansion of limestone, sandstone, and many other rocks may be significantly different for similar rocks from different sources. Coefficients for limestones ranged from 1.2 to 6.5 millionths. The coefficients of all other rocks, excluding the anisotropic materials, quartz, feldspar, and marble, fall within this range. The length change on the return or warming cycle was sometimes slightly different than during the cooling cycle and frequently indicated a higher coefficient. Hockman and Kessler (**1**) of the National Bureau of Standards found this to be particularly true of granite. Kessler (**2**) observed this effect on marble.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 975.

Rock specimens which consist of a single crystal or of a mass of crystals with similar orientation may exhibit anisotropy. Finely crystalline marble had coefficients of −0.9 and 2.0 millionths per deg Fahr on mutually perpendicular axes. Figure 5 shows length changes with temperature for massive crystals of feldspar which had coefficients of 0.5, 1.1, and 9.7 millionths. Quartz had coefficients of 5.6, 6.8, and 8.0 millionths along the various axes. These aggregate minerals, even though saturated, all produced straight normal length-change relationships both above and below freezing. All rocks were tested after soaking overnight.

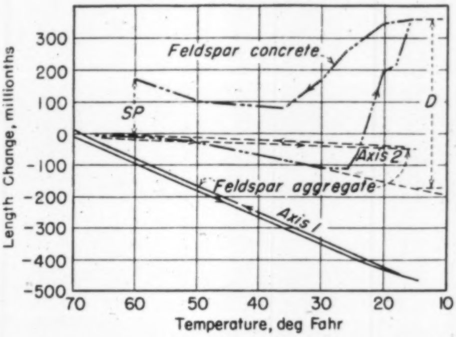

FIG. 5.—Length Change of Concrete and Concrete Aggregate During Temperature Cycle.

Neat Cement:

Neat cement specimens were tested to determine the effect of age, moisture content, fineness, and chemical composition of cement on the thermal expansion (Fig. 6). Continuously moist-cured specimens were tested between 7 and 14 days' age, retested between 45 and 60 days' age, and again between 120 and 130 days' age. The result showed no significant trend with age, so average values are given in Table II. This is in agreement with the findings of Bonnell and Harper (5). Figure 7 shows that the coefficient of thermal expansion increased directly with fineness for six cements ground to different fineness from one clinker. This figure shows that the coefficient was increased about 25 per cent as the fineness of the cement was increased from 1200 to 2700 (Wagner) sq cm per g.

The thermal coefficient of expansion of neat cement is approximately the same for oven-dried and vacuum-saturated specimens, but is considerably higher at intermediate moisture contents. In the range of 80 to 90 per cent saturation the coefficient was increased as much as 100 per cent, as shown in Fig. 8. These results agree with those published by Meyers (6) although the optimum moisture content was not determined. The per cent saturation was based on the assumption that vacuum-saturated specimens were 100 per cent saturated. Computations have

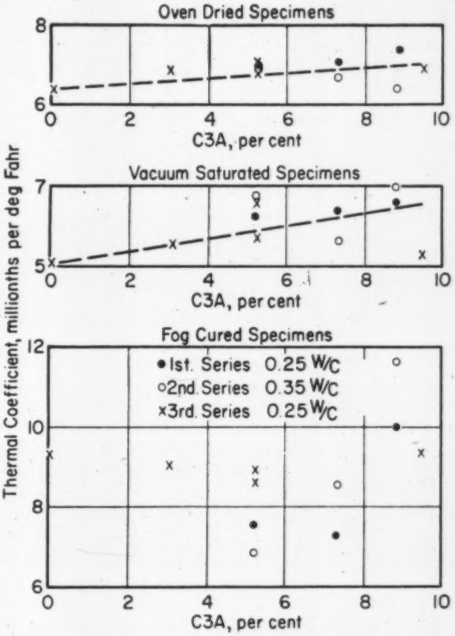

FIG. 6.—Thermal Coefficient of Neat Cement Varied With Moisture Content and Compound C3A.

revealed that this assumption is erroneous in some cases.

A probable explanation for the maximum apparent thermal expansion at optimum moisture content is given by Meyers (6) and Powers (7). Water absorption and swelling pressure of the cement gel at a given relative humidity may vary with temperature and produce an apparent additional thermal expansion. Meyers (6) showed that in very old specimens the amount and effect of optimum moisture content is definitely reduced. The evaporable moisture and gel content was also reduced with age. Autoclaved specimens which supposedly

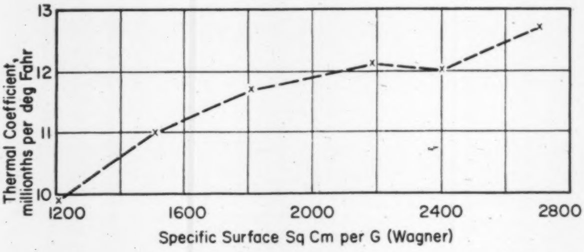

FIG. 7.—Thermal Coefficient of Neat Cement Varied With Fineness.

TABLE II.—THERMAL COEFFICIENT OF NEAT CEMENT SPECIMENS.

Mix	Cement	Fineness, sq cm per g, Wagner	Compound Composition				Water-Cement Ratio	Thermal Coefficient, millionths per deg Fahr			Moisture Conditions		
											Fog Cured		Vacuum Saturated
			C_3S	C_2S	C_3A	C_4AF		Fog Cured	Vacuum Saturated	Oven Dried	H_2O, per cent	Saturated, per cent	H_2O, per cent
No. T-1......	A56	1200	48.8	26.0	8.8	8.0	0.212	9.8	...	...	...	...	...
No. T-2......	A57	1500	48.8	26.0	8.8	8.0	0.220	11.0	...	...	...	...	...
No. T-3......	A58	1800	48.8	26.0	8.8	8.0	0.224	11.7	...	...	...	...	...
No. T-4......	A59	2190	48.8	26.0	8.8	8.0	0.236	12.1	...	...	...	...	...
No. T-5......	A60	2400	48.8	26.0	8.8	8.0	0.236	12.0	...	...	...	...	...
No. T-6......	A61	2700	48.8	26.0	8.8	8.0	0.244	12.4	...	...	...	...	...
No. T-7......	9190	2064	35.0	40.2	7.3	10.7	0.260	11.0	...	...	...	...	...
No. T-8......	9406	1960	36.1	36.2	8.3	13.0	0.240	10.3	...	...	...	...	...
No. T-9......	7488	1735	48.1	25.9	10.5	7.7	0.260	11.3	...	...	...	...	...
No. T-10.....	A777	2015	43.0	32.1	5.2	13.5	0.270	10.7	...	...	...	...	...
No. T-11.....	A777	2015	43.0	32.1	5.2	13.5	0.250	10.1	...	...	...	...	...
No. T-12.....	A777	2015	43.0	32.1	5.2	13.5	0.230	10.6	...	...	...	...	...
No. T-13.....	A109	2028	47.6	30.5	6.1	11.7	0.266	10.1	...	...	...	...	...
No. T-14.....	A109	2028	47.6	30.5	6.1	11.7	0.246	10.7	...	...	...	...	...
No. T-15.....	A109	2028	47.6	30.5	6.1	11.7	0.228	10.6	...	...	...	...	...
No. T-30.....	A777	2015	43.0	32.1	5.2	13.5	0.250	7.6	6.3	6.8	11.11	81.0	13.71
No. T-31.....	9190	2064	35.0	40.2	7.3	10.7	0.250	7.3	6.4	7.0	10.89	82.1	13.27
No. T-32.....	A59	2190	48.8	26.0	8.8	8.0	0.250	10.0	6.6	7.3	9.67	83.9	11.53
No. T-33.....	A777	2015	43.0	32.1	5.2	13.5	0.350	6.9	6.8	6.9	17.83	91.2	19.54
No. T-34.....	A777 + 20 per cent fly ash						0.250	7.1	5.4	6.4	11.18	85.0	13.15
No. T-35.....	A777 + 20 per cent calcined Phillips shale						0.283	6.6	6.5	6.2	14.27	83.9	17.00
No. T-36.....	A777 + 40 per cent fly ash						0.250	7.9	4.6	6.0	10.35	80.6	12.84
No. T-37.....	A777 + 40 per cent calcined Phillips shale						0.342	6.9	5.7	6.2	17.27	81.8	21.12
No. T-38.....	9190 + 40 per cent calcined Phillips shale						0.342	8.0	5.5	5.8	17.56	86.8	20.22
No. T-39.....	A59 + 40 per cent calcined Phillips shale						0.346	8.7	6.1	6.2	18.46	88.9	20.76
No. T-40.....	9190	2064	35.0	40.2	7.3	10.7	0.350	8.5	5.7	6.7	18.28	86.5	21.14
No. T-41.....	A59	2190	48.8	26.0	8.8	8.0	0.350	11.6	7.0	6.4	15.34	83.5	18.37
No. T-42.....	A777	2015	43.0	32.1	5.2	13.5	0.250	9.0	5.7	6.9	10.72	83.0	12.91
No. T-43.....	A1111	2066	41.1	33.8	5.2	13.5	0.250	8.7	6.6	7.1	10.97	89.0	12.32
No. T-44.....	U-3	...	27.3	43.1	3.0	18.2	0.250	9.0	5.6	6.9	12.71	85.3	14.90
No. T-45.....	M1611	...	41.8	32.8	7.4	13.5	0.250	8.8	5.9	7.0	9.46	78.1	12.11
No. T-46.....	M1580	...	74.4	6.3	0	15.5	0.255	9.4	5.1	7.4	7.80	68.4	11.40
No. T-47.....	M1281	...	47.3	27.2	9.4	9.4	0.250	9.4	5.4	6.9	9.52	77.3	12.31

have no gel have been found to have constant thermal expansion at all relative humidities. This indicates that the amount and characteristics of the gel are related to the variation of thermal expansion with changes in moisture content and to the existence of an optimum condition producing maximum thermal expansion.

The author believes that changes in swelling pressures are probably caused by moisture interchanges between capillaries and gel without changing the over-all moisture content. This theory can even explain the fact that vacuum-saturated specimens have the lowest thermal movement of all. When all elements are fully saturated, moisture interchange might be impossible while oven drying leaves nonevaporable water which might still be available for interchange.

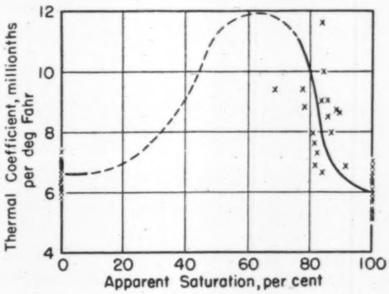

FIG. 8.—Thermal Coefficient of Neat Cement Varied With Moisture Content.

Disruptive expansion of some specimens has been observed during a single cycle of freezing. This action has always been associated with a high degree of saturation. Powers (7) briefly described the gel and capillary structure of hardened cement paste and reported a critical degree of saturation somewhere near 90 per cent of complete saturation. He believes that any concrete critically saturated will fail very quickly in freezing and thawing. The author concurs in this conclusion. The actual percentage may vary slightly due to the elasticity of various constituents in the sample. It will take place when saturation is such that expansion during freezing exceeds the available void space. In such cases the freezing point is depressed to about 20 F, about 4 F below that for concrete under similar conditions, and is indicated by rapid specimen expansion as shown in Fig. 9. This will always occur if the moisture content of the specimen is actually above critical saturation. Later discussion will show that pressures far beyond the possible resistance of cement paste will be developed. The *D* value in Fig. 9 for neat cement and in Fig. 5 for concrete is the expansion due to freezing and is the deviation of the reading at 15 F from the computed straight line extension of the thermal movement before freezing. These figures are similar to data observed by Valore (8).

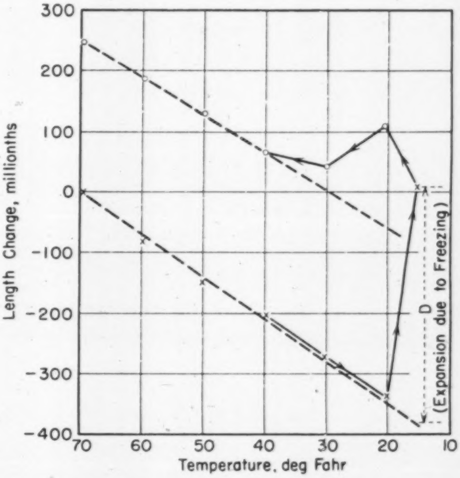

FIG. 9.—Rapid Expansion Caused by Freezing of Water in Saturated Neat Cement.

Many critically saturated specimens give greater coefficients of thermal expansion after freezing than before, while specimens which are not critically saturated tend to give parallel results on successive cycles. A possible explanation of this behavior is that freezing might cause an exchange of water between gel and capillaries to approach more nearly the condition of optimum saturation.

Curves published by other investigators indicate that the thermal coefficient is essentially constant at critical saturation and above. Some evidence of this trend is shown for specimens No. T-33 in Table II. These specimens had essentially constant thermal coefficients of 6.9, 6.8, and 6.9 millionths per deg Fahr at 0, 91.2, and 100 per cent apparent saturation. This indicates that moisture changes from 90 to 100 per cent have little effect on the thermal coefficient. Water appears to be the major factor

TABLE III.—TESTS ON CONCRETES AND MORTARS WITH CONTROLLED AGGREGATE MINERALS.

¾-in. maximum gravel
Sand per cent = 50
Slump = 2 to 4 in.

Mix	Mix Data				Aggregate Description		Thermal Coefficient, millionths per deg Fahr	D
	Cement	Water-Cement Ratio	Parts Aggregate	Air, per cent	Sand[a]	Gravel[b]		
No. C-1	8487	0.51	4.94	...	Angostura natural	Angostura limestone	3.3	48
No. C-2	8487	0.51	4.96	...	Angostura natural	Cherry Creek	5.0	115
No. C-3	8487	0.51	5.20	...	Angostura natural	Buffalo Park quartz	5.5	438
No. C-4	8487	0.51	4.48	...	Angostura limestone	Angostura limestone	2.4	45
No. C-5	8487	0.51	4.14	...	Bridge Canyon limestone	Bridge Canyon limestone	3.4	182
No. C-6	8487	0.51	4.92	...	Local	Bridge Canyon limestone	3.4	66
No. C-8	8487	0.51	4.06	...	Bridge Canyon limestone	Buffalo Park quartz	4.1	150
No. WD 122	2735	0.51	4.75	1.0	Grand Coulee	Grand Coulee	4.0	22
No. WD 123	2735	0.49	4.55	3.4	Georgia marble	Georgia marble	2.3	20
No. WD 124	2735	0.51	4.56	1.1	Buffalo Park quartz	Buffalo Park quartz	5.8	301
No. WD 125	2735	0.51	4.25	1.8	Feldspar	Feldspar	2.9	500
No. WD 126	2735	0.51	4.66	0.8	Buffalo Park quartz	Georgia marble	4.2	552
No. WD 127	2735	0.51	4.40	1.0	Buffalo Park quartz	Feldspar	3.5	1089
No. WD 128	2735	0.51	4.40	1.4	Feldspar	Buffalo Park quartz	3.3	968
No. WD 129	2735	0.51	4.40	1.4	Quartz and feldspar	Quartz and feldspar	4.1	1108
MORTAR MIXES—DO NOT CONTAIN GRAVEL								
No. T-16	A777	0.45	2.25	...	Feldspar	...	4.2	...
No. T-17	A777	0.45	2.25	...	Buffalo Park quartz	...	7.0	...
No. T-18	A777	0.45	2.25	...	Georgia marble	...	4.1	...
No. T-19	A777	0.45	2.25	...	Bridge Canyon limestone	...	4.0	...
No. T-20	A777	0.45	2.25	...	Angostura limestone	...	4.4	...
No. T-21	A777	0.45	2.25	...	Table Mountain basalt	...	5.8	...
No. T-22	A777	0.45	2.25	...	Grand Coulee	...	5.4	...
No. T-23	A777	0.45	2.25	...	Local	...	6.3	...
No. T-24	A777	0.45	2.25	...	Kimball, Neb.	...	6.0	...
No. T-25	A777	0.45	2.25	...	Republican River	...	6.6	...
No. T-26	A777	0.45	2.25	...	Hungry Horse	...	5.9	...
No. T-27	A777	0.45	2.25	...	Bridge Canyon limestone and quartz	...	5.6	...
No. T-28	A777	0.45	2.25	...	Quartz and feldspar	...	5.3	...
No. T-29	A777	0.45	2.25	...	Quartz and Georgia marble	...	5.5	...

[a] Sand grading, per cent

Pan	No. 100	No. 50	No. 30	No. 16	No. 8
5	16	24	25	15	15

[b] Gravel grading, per cent

No. 4 to ⅜ in.	⅜ to ¾ in.
40	60

contributing to changes in thermal expansion of neat cement. Changes from other factors are probably minor except as these factors influence the amount and characteristics of the gel and capillaries, and hence the ability of water to change the thermal expansion.

Mortars and Concretes:

Many mortar and concrete specimens were tested early in the program when it was believed that 28 days of fog curing followed by several weeks' immersion in water would give results closely simulating saturated conditions. The degree of saturation was not determined, but concretes showing high *D* values (Fig. 5), are apparently above critical saturation, while concretes with low *D* values are apparently below critical saturation. The concretes with low *D* values (WD 122 and 123, Table III) had good resistance to freezing and thawing.

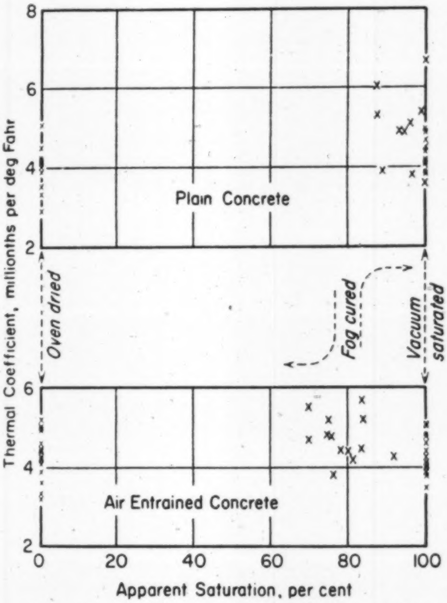

FIG. 10.—Air Entrainment Reduced Moisture Content of Fog Cured Concrete.

The apparent moisture contents of fog-cured concretes are shown in Table IV. Moist-cured plain concretes frequently showed moisture contents of 95 per cent saturation, while air-entrained concretes have much lower percentage of apparent saturation (Fig. 10). These values were computed using oven-dried and vacuum-saturated weights to determine the water content. Air-entrained concretes have a lower percentage of apparent saturation after moist curing and are more difficult to saturate completely even under vacuum.

Generally the thermal coefficients of mortar and concrete are directly related to the coefficients of the aggregate. Mullen (9) suggested that the coefficient of concrete could be computed by using weighted averages of the coefficients of aggregate and neat cement. This would be possible if the correct value for neat cement could be determined.

Several investigators (10, 11) have ably demonstrated an incompatibility between cement mortar and coarse aggregate, which has generally been attributed to wide differences in the coefficients of thermal expansion of the materials. Several combinations of low-coefficient coarse aggregate with high-coefficient mortars are shown in Table III.

About half of these concretes showed large *D* values while the others produced relatively straight length-temperature relationships. Higginson and Kretsinger (12) show correlation between these *D* values and durability for this series of tests. There is a definite indication that other factors are probably more important than thermal incompatibility. All specimens which failed rapidly expanded at some temperature below 32 F. The higher the *D* value the more rapid the deterioration. The moisture content of the mortar or concrete specimen is of prime importance. Specimens above critical saturation failed rapidly.

Theoretically the ice pressure in these tests could approach 9000 psi. Powers (7) showed that 736 psi is necessary to reduce freezing temperature 1 F. Dorsey (13) states that ordinary Type I ice can be formed at pressures up to about 30,000 psi. Obviously some difference must exist in the concrete which either allows supercooling so that ice does not form, or provides escape from the full effects of the possible pressure. No concrete could withstand the pressure if true critical saturation were exceeded. The obvious conclusion is that vacuum saturation does not produce 100 per cent saturation and in some cases may be less than 91 per cent. Probably aggregate porosity, density, water-cement ratio, entrapped and entrained air, and many other factors affect the degree of saturation. Sealed or nearly sealed small voids in air-entrained concrete, which are very hard to fill completely, probably influence moisture interchange. This is indicated by comparing the moisture content of the plain and air-entrained fog-cured specimens (70 to 100 per cent saturated, Fig. 10). The above observation may partially explain the greater durability of air-entrained concrete. The variation of the thermal coefficient with changes in moisture content is much less in concrete than in neat cements (Figs. 8 and 10).

TABLE IV.—THERMAL COEFFICIENTS AND MOISTURE CONTENTS OF VARIOUS CONCRETES.

Mix	Fog-Cured Samples						Vacuum Saturated Samples				Oven-Dried Samples	
	Plain Concrete			Air-Entrained Concrete			Plain Concrete		Air-Entrained Concrete		Plain Concrete	Air-Entrained Concrete
	Coefficient[a]	*D*	Saturated, per cent	Coefficient[a]	*D*	Saturated, per cent	Coefficient[a]	*D*	Coefficient[a]	*D*	Coefficient[a]	Coefficient[a]
No. 1106......	4.9	−67	93.9	4.3	−60	91.6	3.8	−20	4.3	+124	4.2	3.9
No. 1111......	6.0	−5	87.0	5.7	−56	83.1	6.6	−2	5.1	−42	5.1	5.2
No. 1118......	4.9	−18	94.5	4.2	−35	80.8	4.5	−12	4.7	−7	...	...
No. 1127......	4.4	−12	99.4	4.8	−39	74.6	3.8	−25	4.4	−21	...	...
No. 1128......	3.9	−15	88.7	3.8	−19	75.2	4.1	−18	3.5	−35	...	...
No. 1142......	3.8	+7	96.7	2.9	−5	73.3	3.6	+15	3.8	−35	...	...
No. 1167......	4.9	+30	100.0	4.7	−3	69.6	4.2	+316	4.1	+403	4.5	4.4
No. 1179......	...	...	...	5.5	+9	69.3	...	...	4.3	+650	...	4.2
No. 1180......	...	...	...	4.5	+3	83.1	...	...	3.9	+670	...	5.0
No. 1181......	...	...	...	4.4	−3	79.6	...	...	4.0	+1061	...	4.6
No. 1182......	...	...	...	5.2	+10	74.8	...	...	4.1	+415	...	4.3
No. 1196......	5.4	−5	98.7	5.2	−22	83.4	4.9	+37	4.6	−1	4.1	4.4
No. 1207......	...	...	...	5.4	+25	67.8	...	...	5.0	+515	...	...
No. 1213......	...	...	...	4.4	−25	77.8	...	...	4.8	+45	...	4.4
No. 1221......	5.1	+6	95.4	3.8	−24	75.5	4.1	+20	4.0	+26	4.0	5.0
No. 1229......	5.3	+4	87.4	4.8	−45	75.3	4.7	+23	4.8	+9	3.5	4.3

[a] Thermal coefficient is in millionths per deg Fahr.

Temperature length-change curves for concretes below critical saturation are usually good, with only a few points deviating from a straight line. Specimens with moisture contents at critical saturation or above, expand at some temperature below freezing. In concrete this expansion starts between 23 and 27 F, averaging 24 F. This action produces a definite upward surge of the curve (Fig. 5) which does not return to indicate final thawing until the temperature has returned to 32 F or above. Figure 9 shows a curve for neat cement. When the concrete or mortar temperature returns to 70 F, an upward displacement or permanent set is usually evident. This displacement is usually less than the freezing expansion, especially when the movement is large.

CONCLUSIONS

1. Similar rocks from different sources may have totally dissimilar coefficients of expansion.

2. Anisotropy is likely to exist in rocks having crystalline structure, especially if some degree of orientation is present.

3. The principal factor contributing to the thermal coefficient of neat cement is moisture content or per cent saturation of the sample. While many factors, including fineness and chemical composition, contribute to thermal coefficient, their contributions are probably controlled by the degree of saturation of the sample.

4. In neat cement an "optimum" moisture content produces a maximum thermal coefficient. The thermal coefficient of neat cement is minimum for a saturated condition, slightly higher when oven dried, and maximum at some intermediate moisture content. The maximum thermal coefficient of neat cement, for some specimens, is approximately double the minimum value.

5. A specimen above a critical degree of saturation will expand excessively upon a single freezing and break apart in a very few cycles.

6. The coefficient of thermal expansion of concrete can be determined from the weighted averages of the coefficient of thermal expansion of the various constituents if the correct moisture effect can be determined.

7. Durability and thermal coefficient of expansion are both affected by the ease with which concrete becomes saturated and the degree of actual saturation.

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DISCUSSION

Mr. Hubert Woods.[1]—Figure 7 appears to relate the fineness of the cement to the thermal coefficient of expansion of the neat cement bars made from those cements. I presume they were not completely hydrated, were about the same age, and therefore not the same degree of hydration. Would that be a correct assumption to make? A possible explanation for the apparent relationship between the fineness of the cement and the coefficient of expansion might be offered. If the bars were tested at the same age, then there would be in the various specimens different amounts of hydrated cement; therefore the cement specimen tested would be really a composite specimen consisting in part of unhydrated cement and in part of hydrated cement. These two substances have different coefficients of expansion, so what you are measuring would be a composite coefficient of expansion of two different things. I wonder if there was any information to throw light on whether that was the case or whether there is any real effect of fineness on the coefficient of expansion of the completely hydrated cement paste?

Mr. William Van Breemen.[2]—I understand that, in a saturated condition, if the temperature of the concrete falls below the freezing point the concrete will actually expand rather than continue to contract. If this is so, could we then say that in the case of a concrete pavement that has been exposed to considerable rainfall immediately prior to the occurrence of very cold weather, the slabs will at first contract as the temperature lowers, but that when freezing temperature has finally been reached they will then begin to expand?

Mr. H. S. Meissner.[3]—I would agree to that, yes.

Mr. E. C. Higginson.[4]—About 26 F is usually the temperature where they begin to expand.

Mr. Van Breemen.—I take it then that if the change in length were to be plotted graphically, there would be a change in the gradient at about 26 F.

Mr. Higginson.—Below 26 F the gradient would go up.

Mr. C. H. Scholer.[5]—It would seem that in any question of incompatability in relation to thermal expansion of the constituents of concrete, it is equally important that consideration be given to the modulus of elasticity of these components. The internal stress that may develop due to differing characteristics of the components is affected equally by the thermal coefficient of expansion and by the modulus of elasticity. I would like to ask if both of these factors were given consideration in this study.

Mr. Meissner.—Mr. Mitchell made no analysis of those effects. He did not take that into consideration in the paper. There is no discussion of that but certainly such conditions do exist.

In answer to Mr. Van Breeman, you

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will recall that there were negative "*D*" values under some circumstances; this showed that concrete contracted further as the temperature declined. If you would say saturated concrete I would agree with you that concrete usually expands when frozen.

MR. L. J. MITCHELL (*author's closure*).— The assumed conditions which Mr. Woods proposes are correct. However, the amount of water in the mix and, hence, in the final hardened mass is also a factor. I believe that Mr. Meyer found that coefficient of thermal expansion decreased with age and increasing hydration. This would seem to contradict the theory that more complete hydration caused a higher coefficient of expansion in the product. The mixes, T-1 to T-6, inclusive, in Table II would seem to sustain either the water content theory or the fineness theory about equally well. These are the best controlled, most thoroughly uniform series of specimens tested. The cements were all identical ages when mixed, mix conditions were identical as the time was the same, and tests were at approximately the same age. The uniformity control on these cements was also good compared even to laboratory blends, such as A-777, which are used for laboratory tests and comparisons. This later cement, A-777, while being as nearly uniform throughout as is possible to make a large amount of a blend, shows evidence within this table that tests made at different times from different drums of this one cement cannot be depended upon to show a trend in this test. One or two isolated pairs of specimens are now available with control comparable to the above. These specimens show results that are similar to those shown in Fig. 7. However, I do not attribute this effect to any one thing. I believe that it is caused by those factors which go with differences in fineness.

Mr. William Van Breemen's assumptions would be correct if an appreciable portion of the concrete was above critical saturation or about 91 per cent.

Commenting on Mr. C. H. Scholer's remarks, it seems unlikely that thermal stresses could produce the extreme magnitudes of movement shown for feldspar concrete in Fig. 5, or that this same action would take place in neat cement, as shown in Fig. 9. Expansion due to the formation of ice could account for the observed expansions.

I cannot fully explain the negative "*D*" values mentioned by Mr. Meissner. One theory says that since the coefficient of expansion of ice is much greater than that of the other materials present in the concrete, it will (where it does not expand enough to disrupt the surrounding material) act as an additional aggregate of higher coefficient and thus cause the change in slope of the curve after passing the temperature where freezing takes place.

SIGNIFICANCE OF TESTS FOR CHEMICAL REACTIONS OF AGGREGATES IN CONCRETE*

By William Lerch[1]

Synopsis

The chemical reactions involving aggregates in concrete may be beneficial or deleterious depending on the nature of the reactions. The chemical reactions that increase the bond between the aggregate and the matrix may be beneficial, while those that cause abnormal expansion and consequent map cracking are deleterious. There are no direct tests to evaluate the beneficial reactions, but results obtained by some performance tests, together with observations made in connection with the tests, give some indication of the bond between the aggregate and the matrix. Extensive studies have been made in recent years of the deleterious reactions of aggregates in concrete that can cause abnormal expansion. The Society has adopted three tentative methods of tests for aggregates to determine their potentiality for producing abnormal expansion in concrete. Additional methods of test have been described or are being studied. Some of these tests can be used also for the study of reactive siliceous admixtures or other inhibitors that may reduce or eliminate the expansion. This paper discusses the significance of these various tests for chemical reactions of aggregates in concrete.

Beneficial Chemical Reactions

The bond between the aggregate and the matrix has a significant effect on the strength, permeability, and durability of concrete. It is probable that the bond is affected by chemical reactions at the surface of the aggregate and by the surface texture or other characteristics of the aggregate. There is no suitable direct method available for measuring the bond between the aggregate and the matrix. The strengths obtained in testing the mortar-making properties of fine aggregates (ASTM Method C 87 – 52),[2] the compressive strength of concrete cylinders (ASTM Method C 39 – 49),[3] and flexural strength of concrete (ASTM Method C 78 – 49),[4] and visual observations made in connection with these tests provide some information about the bond. High strengths are some indication of a good bond. When it is observed that breaks occur through the aggregate, there is evidence of good bond. When the break occurs around the aggregates and they can be pulled out of their sockets, there is evidence of a poor bond. The entire problem of the chemical and physical factors that influence the bond between aggregate and matrix is one that deserves further study.

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[2] Method of Test for Measuring Mortar-Making Properties of Fine Aggregates (C 87 – 52), 1952 Book of ASTM Standards, Part 3, p. 939.

[3] Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 49), 1952 Book of ASTM Standards, Part 3, p. 1040.

[4] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 49), 1952 Book of ASTM Standards, Part 3, p. 1051.

DELETERIOUS CHEMICAL REACTIONS

The recognition of deleterious chemical reactions of aggregates in concrete is relatively recent. Prior to about 1940, most aggregates were usually considered to be chemically inert as used in concrete. The publication in 1940 of papers by Thomas E. Stanton on "Influence of Cement and Aggregate on Concrete Expansion" (**1**)[5] and "Expansion of Concrete Through Reaction Between Cement and Aggregate" (**2**) clearly demonstrated that certain reactive silica constituents, present in some aggregates, can react with the alkalies in cements to cause abnormal expansion and map cracking in mortars and concretes. These observations led to an intensive study in many different laboratories of the entire problem of chemical reactions of aggregates in concrete. These studies confirmed the original finding by Stanton that the alkali-aggregate reaction can cause abnormal expansion in concrete, and they have indicated also that there probably are other, not yet clearly defined, chemical reactions of aggregates in concrete that can cause abnormal expansion. The studies have been directed also toward developing: (1) methods of test for aggregates to determine their potentialities of producing deleterious chemical reactions in concrete, and (2) methods of tests for inhibitors including reactive siliceous admixtures that minimize or eliminate the abnormal expansion resulting from the reactions.

TESTS FOR DELETERIOUS REACTIONS

The significance of the various tests for deleterious reactions of aggregates in concrete will be discussed.

Field Service Records:

The service records of concrete structures provide the best information for the selection of aggregates. When an aggregate has been used extensively, with cements of different compositions, and the structures have good service records, it can be assumed that the aggregate does not contain material that produces deleterious chemical reactions in concrete. However, it is often necessary to use aggregates from new sources of supply, or in different combinations, where such service records are not available. It then becomes necessary to make laboratory tests to determine the potential reactivity of the aggregates.

Mortar Bar Test:

Beginning with the work by Stanton in 1940, followed by that of a number of other investigators, a mortar bar test for potential reactivity of cement-aggregate combinations was evolved. In 1950, it was issued as an ASTM Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227). The scope of this test states that:

> This method of test is intended to determine the potential expansive alkali reactivity of cement-aggregate combinations by measuring the expansion developed by the combinations in mortar bars during storage under prescribed conditions of test.[6]

It can be used for both the fine and coarse aggregate. The fine aggregates are tested in a grading meeting the requirements of the specifications for the project. The coarse aggregates are crushed, sieved, and recombined to a grading specified for the test. The test has been used extensively by a number of different laboratories and is usually considered to be the most reliable test now available for determining the potential alkali reactivity of aggregates. It can be used also for studies of inhibitors that reduce or eliminate the expansion. The disadvantage of this method is that it requires several months to obtain conclusive results.

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[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 988.

[6] 1952 Book of ASTM Standards, Part 3, p. 44.

The ASTM Tentative Specifications for Concrete Aggregates (C 33 - 52 T), contain the following note for both fine and coarse aggregate:

> Fine (coarse) aggregates producing excessive expansions, when tested for potential alkali reactivity in accordance with the Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (ASTM Designation: C 227), contain injurious amounts of reactive materials. Fine (coarse) aggregates that have shown harmful reactions in concrete generally have produced expansions of more than 0.05 per cent at 6 months or 0.10 per cent at 1 yr when tested with a cement containing alkalies in excess of 0.8 per cent expressed as sodium oxide. However, aggregates that produce expansions of more than 0.05 per cent at 6 months but less than 0.10 per cent at 1 yr should not be expected to be harmful.[7]

Stanton (2) made up mortar and concrete bars of California (Oro Fino and Coyote) fine and coarse aggregates and then subjected some of these bars to continual wetting, some to continual dry exposure under normal laboratory temperature and humidity conditions, and some to alternate wetting and drying. In some cases the wetting-and-drying cycle involved drying in an oven at 150 F and soaking in water at 70 F and drying at 150 F and soaking in water at 160 F. No excessive expansion was observed under any of these conditions of test even though pavements constructed with these aggregates had developed excessive expansion and map cracking. In the meantime, however, when the cover was removed from a 2 by 4-in. mortar cylinder which had been cast and retained in a tin container for one year, the specimen was observed to be covered with blotches fringed with a white efflorescence, and in a short time the entire specimen became covered with cracks similar to those observed in pavements and structures in the area under investigation. It was apparent that when the specimens were kept in sealed containers, or at least protected from any drying out, but at the same time prevented from any leaching of salts by immersion in water, a chemical reaction was taking place which caused an excessive expansion of the mortar. These observations led to the procedure of storing the mortar bars over but not in contact with water, in tightly sealed metal containers (ASTM Method C 227).[6]

Figure 1 illustrates the expansion of sand-cement mortars in the mortar bar test as influenced by the type of sand and the alkali content of the cement. The six cements used in these tests had total alkali contents expressed as Na_2O ranging from 0.45 to 1.14 per cent. These results, reported by Stanton (2), show that the Monrovia sand is not reactive and does not develop excessive expansion with any of the cements. The Salinas River and Oro Fino sands are reactive, and with these sands the expansion appears to be related to the alkali content of the cement and is not excessive when the sands are used with a low-alkali cement in this combination.

A number of investigators (3-6) have studied the effect of storage temperature on the expansion obtained with the mortar bar test in an attempt to accelerate the test. The results of these studies have shown that the expansion occurs more rapidly and is usually higher for specimens stored in the covered containers at 100 F than that of companion specimens stored at 70 F or at temperatures slightly higher than 100 F. These observations led to the adoption of the storage temperature of 100 ± 3 F in ASTM Method C 227.[6]

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[7] 1952 Book of ASTM Standards, Part 3, p. 898.

by the Portland Cement Assn. As a result of these findings the method for determining the flow of the mortar was modified in ASTM Method C 227[6] to permit the use of a higher amount of mixing water.

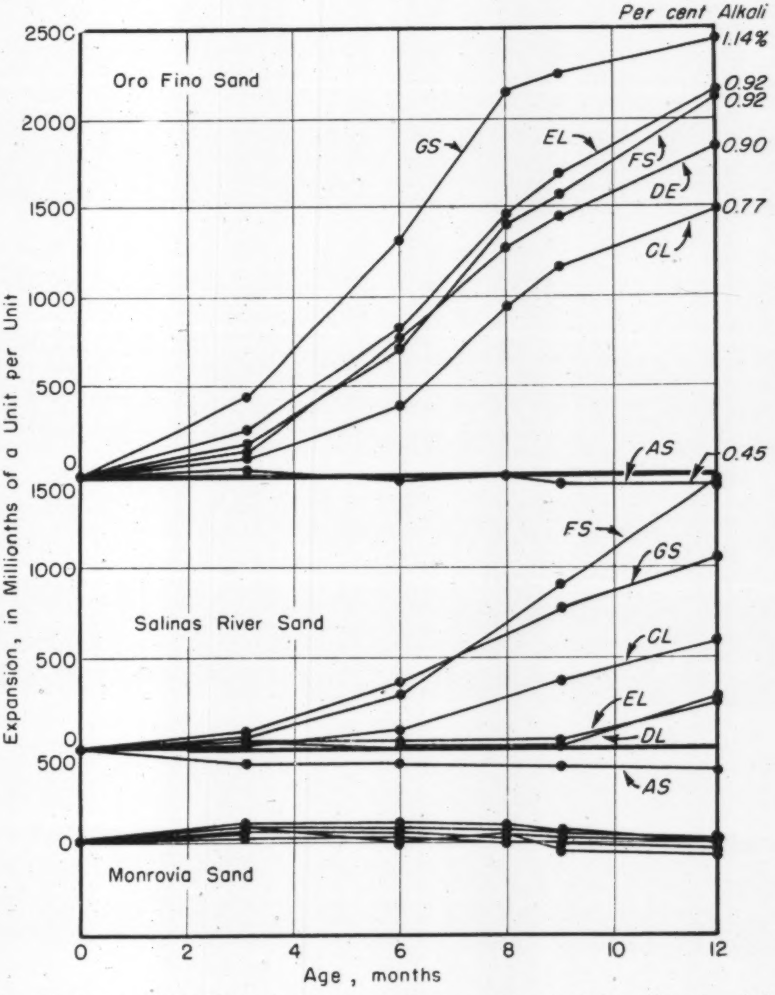

FIG. 1.—Expansion of Sand-Cement Mortars as Influenced by the Type of Sand and the Alkali Content of the Cement.

From paper by Thomas E. Stanton (2).

Stanton (7) reported that the rate of expansion of the mortar bars may be materially accelerated by increasing the percentage of mixing water used in the preparation of the mortars. these observations were confirmed by tests made

The subcommittee on Durability of Concrete, ASTM Committee C-9 on Concrete and Concrete Aggregates, conducted a cooperative series of tests to determine the reproducibility of results obtained in the mortar bar test for potential alkali reactivity of cement-aggregate reaction. The results of these tests were published in 1946 (8). Other investigators have reported on results obtained by using the test (9–16). It has been found, in most cases, that when natural reactive aggregates are used with different cements the expansion increases with increasing alkali content of the cement. However, Hanna (10) and Woolf (16) obtained excessive expansion with low-alkali cements when small amounts of a highly reactive opaline material were used in combination with a neutral aggregate; results of this type have not been reported in tests using natural-occurring aggregates.

There appears to be a very good correlation between results obtained with the mortar bar test and the performance of the aggregates in pavements and structures. Stanton (17) has reported that it is seldom possible to establish as positive a correlation between laboratory tests and field experience as has been established by the mortar bar test (ASTM Method C 227).[6] He lists a number of typical cases of failure of concrete structures through an adverse reaction between cement and aggregate where the field experience was in each case subsequently checked by laboratory tests. In other cases when laboratory tests indicated potential reactivity, subsequent field inspection confirmed the suspected activity in structures in which the same aggregates had been used with a high-alkali cement. Tremper (18), and Mielenz and Witte (12) and Blanks and Meissner (19) have reported similarly good correlations.

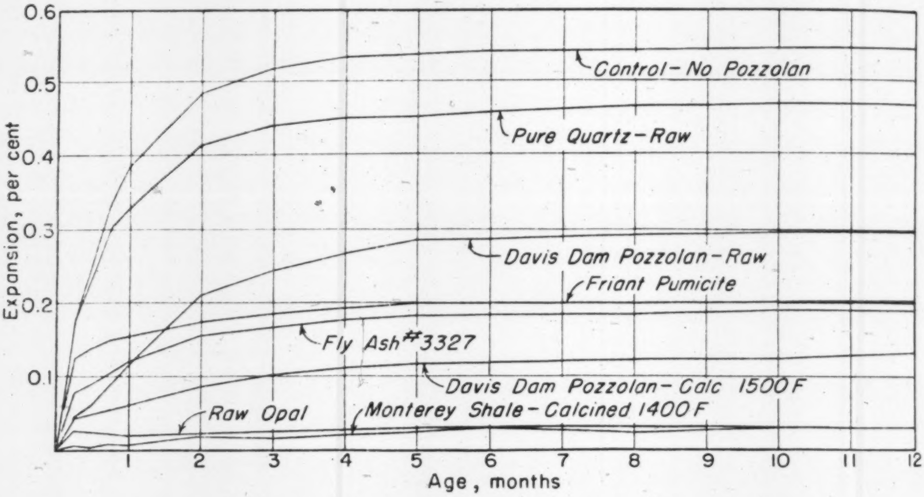

FIG. 2.—Pozzolans Variously Reduce the Expansion of Reactive Mortar.

From paper by H. S. Meissner (22).

1 by 1 by 10 in. bars. 1:2.25 mortar made with high alkali cement (1.20 per cent Na_2O, 0.04 per cent K_2O) and crushed pyrex glass sand. Pozzolans replace 20 per cent by weight of the cement. Specimens stored sealed with moisture at 100 F.

The mortar bar test can be used also for studying inhibitors that reduce or eliminate the expansion associated with the cement-aggregate reaction. A number of investigators have used the mortar bar test to study inhibitors (10, 15, 22–27). Certain calcined reactive siliceous materials have been very effective in reducing the expansion of the mortars as illustrated in Fig. 2, and some of them have been used with satisfactory results in large dams (22, 26). Although a naturally occurring reactive aggregate could be used for such tests, a more uniform supply of reactive material would be desirable. The Bureau of Reclamation has recommended the use of pyrex glass, crushed, screened, and recombined to a specified grading for this purpose (20–21).

Petrographic Examination of Aggregates:

The microscope provides a useful tool for identifying the mineral constituents in aggregates. The petrographic examination of aggregates assumed a rôle of increased importance when it became recognized that the chemical reactions of aggregates in concrete may have a significant effect on the durability of concrete. The Society has adopted a Tentative Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295 - 52 T).[8] It outlines procedures for the petrographic examination of samples representative of materials proposed for use as aggregates in concrete.

TABLE I.—MINERALS DELETERIOUSLY REACTIVE WITH HIGH-ALKALI CEMENTS.

Reactive Minerals	Chemical Composition	Physical Character
Opal	$SiO_2 \cdot nH_2O$	Amorphous
Chalcedony	SiO_2	Cryptocyrstalline Fibrous
Tridymite	SiO_2	Crystalline

A number of papers have been published that show the value of petrographic examinations as a means of identifying potentially reactive minerals in aggregates and the source of reactive materials and the characteristics of reaction products in affected mortars and concretes (2, 3, 28, 31, 32). Some minerals that may be deleteriously reactive with cements (9) are shown in Table I.

Chemical Tests for Reactive Aggregates:

A number of laboratories have studied chemical tests for use in determining the potential reactivity of aggregates. As a result of these studies the Society accepted the Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 - 52 T). "This method of test covers a chemical method for determining the potential alkali reactivity of aggregates and their potentiality for producing abnormal expansion in concrete when used with high-alkali cement. It is based on the amount of reaction of the aggregate with a sodium hydroxide solution under controlled laboratory conditions of test."[9]

The test is based on extensive studies by Mielenz, Greene, and Benton (33). The potential reactivity of an aggregate is measured by the amount of silica dissolved by a 1*N* sodium hydroxide solution from a representative sample of aggregate crushed to the No. 50–No. 100 size, and the concomitant reduction in alkalinity of the solution. It is a rapid test. The samples can be prepared, the test run, and the necessary chemical analyses completed in three working days. It is an empirical or experience test. The authors tested a large number of aggregates by the chemical method and used the same aggregates in combination with a high-alkali cement in the mortar bar test, ASTM Method C 227.[6] They then plotted the results of the chemical test, silica dissolved *versus* reduction in alkalinity, and drew a line through the diagram to separate the innocuous aggregates, those showing less than 0.1 per cent expansion at one year in the mortar bar test, from the reactive aggregates, those showing more than 0.1 per cent expansion, as shown in Fig. 3.

Since a large number of aggregates were used in the preparation of this diagram, it is assumed that other aggregates can be identified as innocuous or reactive by subjecting them to the chemical test, plotting the results in a diagram similar to Fig. 3, and using the same line of demarcation. Results obtained with the chemical test have been discussed in other publications (**12, 14, 15, 34**). The results can be used to identify the presence of reactive material in the aggregate, but they do not always give assurance that it is present in the proportion required to cause abnormal expansion.

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[8] 1952 Book of ASTM Standards, Part 3, p. 978.

[9] 1952 Book of ASTM Standards, Part 3, p. 943.

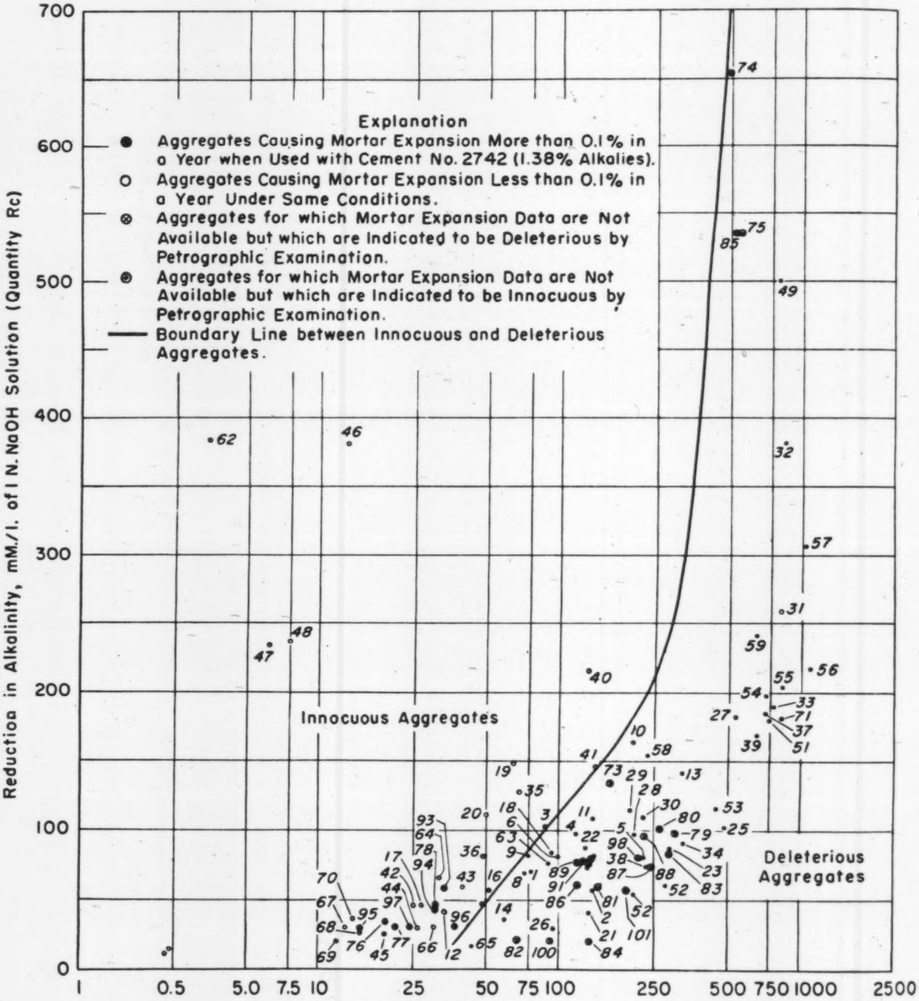

FIG. 3.—Results of Chemical Test with NaOH Solution.

From paper by Richard C. Mielenz and Leslie P. Witte (**12**).

A Wetting-and-Drying Test for Predicting Cement-Aggregate Reaction:

Scholer and Gibson (**35**), and Scholer (**36**) have described a heating-drying and cooling-soaking test for studying deleterious cement-aggregate reactions. It is a modification of a test used earlier by Gibson (**37**). The test provides for exposing saturated specimens of concrete to a drying condition at 130 F for 8 hr, followed by immersion in water at an initial temperature of 70 to 80 F for 16 hr. The cycle is repeated 6 days each week, the specimens remaining immersed over Sunday. The percentage expansion developed by the test specimens provides a measure of the potential cement-aggregate reaction. An expansion of 0.07 per cent or more at one year (285 cycles) indicates a reactive cement-aggregate combination. The results obtained with this laboratory test are in good agreement with the performance of comparable specimens in long-time outdoor exposure and with the performance of similar materials when used in pavements and structures. The type of results obtained with this test are shown in Fig. 4 where Kaw River, Kans., sand-gravel aggregate was used in combination with 24 different cements for these tests.

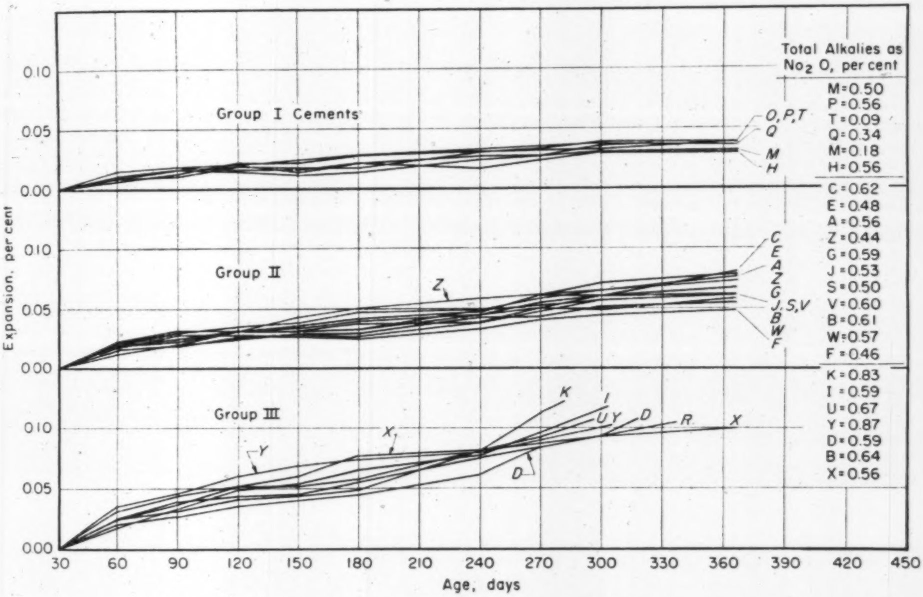

FIG. 4.—Expansion of Concretes Made with Kaw River, Kansas, Aggregate and 24 Different Cements.

From paper by C. H. Scholer and W. E. Gibson (**34**).

The wetting-and-drying test can be used also for studying inhibitors that can be used to reduce or eliminate the abnormal expansion resulting from a deleterious cement-aggregate reaction. Figure 5 shows how additions of neutral coarse aggregates reduce the expansion that occurs with Republican River, Neb., sand-gravel aggregate. Other publications have described the use of this test for studying pozzolans (**38, 39**).

The Mason Jar Test:

Woolf and Smith (**40, 41**) proposed the Mason jar test as a rapid qualitative method of testing mortar or concrete for alkali-aggregate reaction. The interior surface of the jar is coated with a chlorinated rubber or vinyl resin base paint to prevent a reaction between the glass and the concrete. The jar is then filled to the neck with concrete, the cap fastened, and the concrete allowed to harden. A small amount of water, about 25 ml, is then added, the cover replaced, and the jar is set aside for observation. Expansive reactions within the concrete crack the Mason jar. The time required to crack the jar and the number of cracks provides a measure of the chemical reaction of the aggregates in the concrete. The authors claim that a period of test of 28 days appears to be sufficient for the detection of aggregates which may be considered too susceptible to reaction with high-alkali cement to warrant their use in concrete and that the method is suitable for use in the field, as the equipment necessary is inexpensive and can be obtained readily. The test can be used also to study inhibitors that may prevent the excessive expansion. Walker (40) concluded from similar tests that some significant trends are indicated, although clear-cut relationships are not shown. He expressed doubt about assigning to a field test so important a responsibility as determining the chemical reactivity of aggregates.

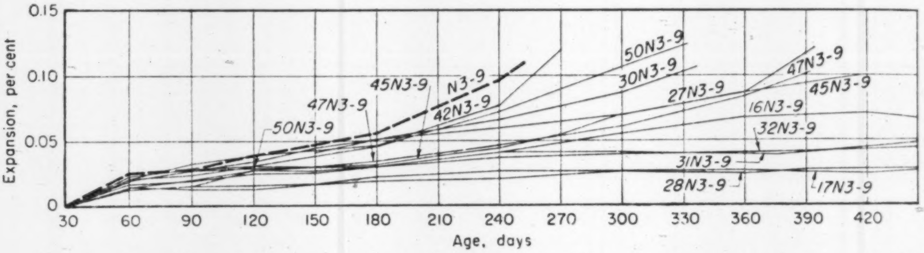

FIG. 5.—Expansion of Concretes Made with Republican River, Nebraska, Sand-Grave (N 3-9) and Additons of Coarse Aggregates.

The heavy dash line N 3-9 represents the expansion of the sand-gravel aggregate concrete. From paper by C. H. Scholer and W. E. Gibson (34).

Volume Change of Cement-Aggregate Combinations Subjected to Variations of Temperature and Water Saturation:

Conrow (42) has described a mortar bar test for detecting excessive volume change of cement-aggregate combinations subjected to variations of temperature and water saturation. For this test the mortar bars are first cured in the molds one day and in water under prescribed conditions at standard temperature for 27 days. They are then stored in water for 7 days at 130 F followed by storage in a drying oven for 7 days at 130 F. The specimens are then returned to continuous water storage at normal temperatures. The expansion of the test specimens is taken as an indication of the relative potential reactivity of the cement-aggregate combination. Special containers are required for storing the specimens in a minimum amount of water to reduce the leaching of soluble salts from the test specimens. Tests of a large number of cement-aggregate combinations were reported. A considerable number of aggregates used in these tests developed excessive expansion. From a study of the data, Conrow concluded that the abnormal expansion cannot be definitely related to any one cement constituent but rather to the following items:

1. The quantity and rate of release of calcium hydroxide during the hydration of the cement,
2. The alkali content of the cement,
3. The fineness of the cement, and
4. The compound composition of the cement.

The excessive expansion, as developed by this test, can be prevented or greatly decreased by the addition of a suitable pozzolanic material. It has not been shown that the results obtained in this test are related to the performance of similar cement-aggregate combinations when used in pavements or structures.

Subcommittee II b, Chemical Reactions of Aggregates in Concrete, of Committee C-9, has under way a coöperative series of tests to determine the reproducibility and significance of the test procedures proposed by Scholer and Gibson and by Conrow. One of the important results of these tests was the observation that relatively small changes in aggregate combinations resulted in a pronounced difference in the expansion of the test specimens.

Miscellaneous Tests:

A number of additional tests have been described. Although it has not been shown that the results obtained by these tests bear any relation to the performance of the aggregates in concrete, they will be reported here to complete the record.

Parsons and Insley (**28**) described a test for determining the reactivity of an aggregate which consists of microscopic examination of polished surfaces of rocks and minerals after etching in various alkali hydroxide and sulfate solutions.

Runner (**43**) described a pat test for observing the chemical reactions of aggregates in concrete. For this test, pieces of aggregate are placed in the bottom of the mold and covered with a neat cement paste. The pats are then examined periodically for the development of a whitish reaction product on the surface which is assumed to result from the reaction of the alkalies in the cement and certain ingredients in the aggregate.

Bean and Tregoning (**44**) tested the reactivity of aggregates by immersing them in alkali hydroxide solutions under definite conditions of concentration and temperature. Weight loss of the aggregate in the test is a measure of its reactivity. Other studies (**33**) have shown that the solubility of an aggregate in alkali solutions alone is not a significant indication of its potential reactivity.

Alderman, Gaskin and Vivian (**45**) have described a qualitative test for cement-aggregate reaction. The test is based upon the distortion produced in a body undergoing a differential expansion. The test specimen consists of a thin bi-aggregate slab of cement mortar in which a material known to be nonreactive is used as aggregate on one side and the aggregate to be tested is used on the other. The first indication of a reaction is given by the development of damp-looking spots on the surface of the mortar. Expansion is shown by warping of the test specimens. Results obtained with the test are reported to be in good agreement with those obtained with the mortar bar test.

CONCLUSION

Progress has been made in developing methods of test for aggregates to determine their potentialities of producing deleterious chemical reactions in concrete. Some of these tests can be used also for the study of reactive siliceous admixtures or other inhibitors that may reduce or eliminate the expansion. However, there is an urgent need for further study of test procedures. Some of the most reliable methods currently available require such a long time to provide significant results that they are not suitable for use as acceptance tests. The rapid tests provide valuable information that can be used to identify the presence of reactive constituents in the aggregate, but they do not always give assurance that the reactive material is present in the proportion required to cause abnormal expansion.

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DISCUSSION

Mr. Bryant Mather.[1]—Mr. Lerch made reference to beneficial and deleterious reactions, and in a later part of his paper he made reference to deleterious and innocuous aggregates. The point that I would like to make is, that we need more information about discriminating between beneficial, innocuous, and deleterious reactions and aggregates.

At the present time we can only classify the reactions with which we are dealing and the aggregates in which we are interested, in terms of their behavior in concrete. If the concrete is harmed by the presence of the aggregate or by the fact that a given reaction took place, then the aggregate is deleterious and the reaction is deleterious.

I think, however, that it may very well be that a particular chemical reaction involving a particular class of aggregates may, if it goes to a limited extent, be beneficial; but if that same reaction, involving those same aggregates, goes further, it may be deleterious. I suggest this, because there are certain preliminary observations which we have made in our laboratory which makes me suspect that this may be the case.

I rather hesitate to be specific, but in order to give an example of the sort of thing that I am speaking of, I would like to mention the chalcedonic chert aggregates of the Gulf Coast, particularly as they occur in parts of Mississippi, Alabama, Louisiana, and Texas. The quick chemical tests involving the determination of solubility and reduction of alkalinity in sodium hydroxide indicate that these aggregates are almost uniformly "deleterious." Mortar-bar tests often indicate abnormal expansion. There are, however, very few service records which indicate that concrete made with these aggregates, even with high-alkali cement, has given trouble in service.

Examination of samples of this concrete indicates that the expected chemical reaction has taken place, but, and this appears to be characteristic, the typical alkali-aggregate reaction in those particular concretes seems to have been beneficial. Mr. Lerch, at the outset of his paper, spoke of the beneficial reaction which improves the bond between the aggregate and the matrix. There are indications that the alkali-aggregate reaction, if it goes only so far, but not too far, does just that. I suggest, therefore, that we must consider these things in terms of degree, as well as kind, when we separate the beneficial from the deleterious, and the innocuous from the deleterious, when we classify both reactions and aggregates.

Mr. Wilson C. Hanna.[2]—It was noticed in Fig. 1 presented by Mr. Lerch that tests were conducted only up to one year and that some specimens had little expansion. My experience has been that where the specimens are examined at the age of several years the expansion with low-alkali cement may be as high as or higher than with the high-alkali cement specimens. It is well to make a note of that. I believe that one cannot depend on cements with as little as 0.4 per cent, or even 0.1 per cent in some cases, total alkali overcoming the unfavorable results.

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PREDICTION OF CONCRETE DURABILITY FROM THERMAL TESTS OF AGGREGATE*

By E. C. Higginson[1] and D. G. Kretsinger[2]

Synopsis

Data are presented on the effect of freezing and thawing, wetting and drying, and other simulated weathering tests on various concretes containing either natural aggregate or aggregate composed of individual rocks and minerals. The effect of thermal coefficient and diffusivity of these aggregates on the deterioration occuring during these tests is discussed, but no correlation is found between the test results and the thermal properties of the materials. Evidence is presented showing that the expansion occuring in saturated concrete during the measurment of coefficent of expansion can be correlated with freezing-and-thawing test results, thus providing a quick method of predicting durability of concretes.

In recent years, considerable thought and effort have been directed toward the identification of the causes of concrete cracking, spalling, and general disintegration. In some cases, deterioration has been attributed to differences in thermal properties of the coarse aggregate, sand, and cementing paste. The thermal properties of concrete materials that are judged possible of causing distress are specific heat, conductivity, diffusivity, and coefficient of expansion. Since diffusivity (**1**)[3] is related directly to specific heat and conductivity, only variations in diffusivity and thermal coefficient are considered likely causes of distress.

When considerable differences exist between thermal coefficients of the coarse aggregate and the mortar or cement paste, stresses may exist that are theoretically sufficient to disrupt the concrete. This may also be the case where the coarse aggregate has a diffusivity value considerably different from the matrix. Such stresses have been computed by various investigators (**2**). The Bureau of Reclamation is trying to determine the cause of unexplained concrete failures in the Kansas-Nebraska area.

The tests reported in this paper were performed to determine whether or not the thermal variations in the weathering tests were the cause of deterioration. Two analyses were made; the first to determine the relationship between thermal properties and the deterioration caused by various weathering tests. The second is concerned with the correlation of freezing-and-thawing durability with expansions that occur during thermal measurements such as those used by Valore (**3**) of the National Bureau of Standards and Mitchell (**4**) of the Bureau of Reclamation.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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[2] Former Materials Engineer, Bureau of Reclamation, Denver, Colo.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1000.

TEST PROCEDURE

Aggregates composed of individual rocks and minerals, including quartz, limestone, basalt, marble, feldspar, and some natural aggregates that had a wide range of thermal property values were selected for these tests. In addition, tests were made on concrete containing polished and etched steel balls as a coarse aggregate. The specific heat, conductivity, diffusivity, and thermal coefficients of these aggregates were determined. Thermal coefficients ranged from zero to nine millionths and diffusivity constants from 0.029 to 0.104 ft squared per hr. Concrete specimens were made containing each of these aggregates; however, all specimens were not subjected to the same weathering cycle.

All concretes made for this investigation contained $\frac{3}{4}$-in. maximum size aggregate and had water-cement ratios of 0.51 by weight. Water and cement contents were varied slightly from mix to mix to give slumps between 2 and 3 in. Slump, unit weight, and air content determinations were made on all fresh concretes. Each concrete mixture was dry batched in the mixer before adding the mix water, after which it was mixed for 5 min in a revolving drum-type mixer. Specimens included 3 by 3 by 15-in. bars and 3 by 6-in. cylinders for weathering cycles, 8 by 16-in. cylinders for specific

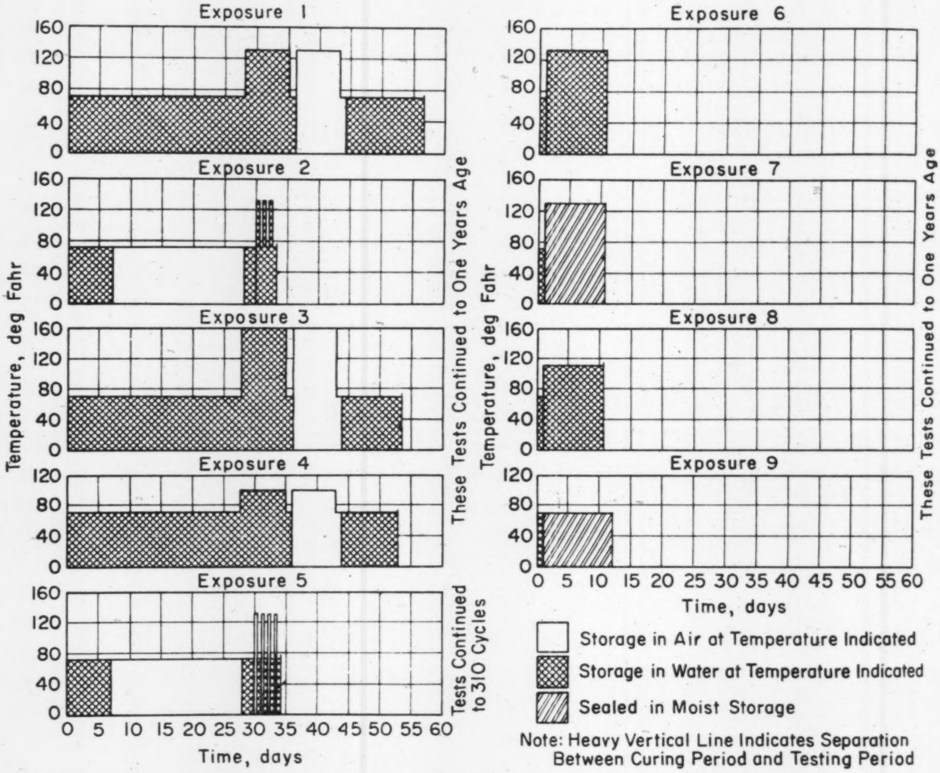

FIG. 1.—Simulated Weathering Exposures Showing Temperature and Moisture Variations.

heat, conductivity, and diffusivity tests, and 1 by 3 by 3-in. prisms cut from bars for thermal coefficient of expansion determinations. The thermal coefficient of expansion was determined on individual pieces of aggregate and on the small concrete prisms. Length changes were determined with an electric strain gage (4) sensitive to 1 microinch as the specimens were passed through a temperature cycle of 70 to 15 F and back to 70 F in about an 8-hr period. The length changes were obtained at 5-day intervals and provided valuable information on volume changes of saturated concrete.

TABLE I.—DESCRIPTION OF AGGREGATES.

Laboratory Designation	Aggregate	Source	Description
8322	Crushed quartz	Buffalo Park, Colo.	Massive, milky, pegmatitic. Pure quartz
M-945	Crushed feldspar	Buffalo Park, Colo.	Microcline-perthite feldspar
M-439	Crushed marble	Georgia	
M-675	Natural sand and gravel	Brett pit, Coulee Dam, Wash.	Mainly basalt, granite, and rhyolite rocks with less abundant quartzite, sandstone, and mica schist
M-145	Natural sand and gravel	Walt Rodman pit, Kimball, Neb.	Gravels: granites, gneisses, schist, quartz, quartzites, and limestones Sand: same as gravel with pink feldspar
M-149	Natural sand and gravel	Davidson pit, Indianola, Neb.	Gravels mainly granites and feldspar. Sand mainly quartz and feldspar. Reactive with alkalies in cement
Sample C-8	Crushed limestone	Bridge Canyon Dam site, Ariz.	Hard, dense, medium- to fine-grained limestone
7108	Crushed limestone	Angostura Dam site, S. Dak.	White, massive, firm, fine-grained limestone
7406	Natural sand	Angostura Dam site, S. Dak.	Mainly sandstone, limestone, and quartz
8085	Natural sand	Cherry Creek, Colo.	Mainly quartz and feldspar
8185	Gravel	Clear Creek, Colo.	Mainly granite, granite gneisses, and diorite
2037	Crushed siliceous magnesian limestone	Monterey County, Calif.	Siliceous magnesian limestone

All concrete specimens were stored in a fog room at 73 F and 100 per cent relative humidity for 24 hr immediately after casting. Subsequent curings and test procedures in the various simulated weathering cycles are shown graphically on Fig. 1. Exposures 1 and 2 were suggested by A. D. Conrow of the Ashgrove Lime and Portland Cement Co. Exposures 3 and 4 are variations of Exposure 1. Exposure 5 was designed by C. H. Scholer (5) of Kansas State College. Exposures 6, 7, 8, and 9 were designed by the laboratory staff of the Bureau of Reclamation. Temperatures in these weathering cycles vary from 70 to 160 F. In some of the exposures there are variations in temperature during the curing periods and in others the temperature variations occurred during the test period. In some cases these variations were accomplished by the use of water, and in others air was used as the heating or cooling medium.

In addition to the weathering cycles, some specimens were subjected to accelerated freezing and thawing (6) in both water and in air. The temperature range of the freeze-thaw cycle was 10 to 70 F, a complete cycle being made in 3 hr at the rate of 50 cycles per week.

Specimens shown in Fig. 5 were subjected to a total of 125 alternations of either 70 F, 50 per cent relative humidity storage, 40 to 100 F, or −10 to 100 F temperature changes in a dry condition prior to being subjected to accelerated freezing and thawing in air. This latter temperature alternation (−10 to 100 F) was used solely to obtain a 110 F temperature differential.

TABLE II.—THERMAL PROPERTIES OF CONCRETES AND AGGREGATES.

Specimen	Specific Heat, Btu per lb deg Fahr	Conductivity, Btu per ft hr deg Fahr	Diffusivity, sq ft per hr	Density, lb per cu ft	Thermal Coefficient, millionths		
					Axis 1	Axis 2	Axis 3
Concrete Containing							
M-675 Basalt[a]	0.246	1.105	0.0298	150.8	3.95		
M-439 Marble	0.255	1.216	0.0315	151.4	2.32		
8322 Quartz	0.232	2.392	0.0698	147.8	5.78		
M-945 Feldspar	0.246	1.152	0.0323	145.0	2.93		
7108 Limestone	0.242	1.302	0.0360	149.1	2.44		
C-8 Limestone	0.234	1.691	0.0462	156.5	3.38		
Aggregate							
M-675 Basalt[b]	0.204	0.990	0.0288	168.2	3.24		
M-439 Marble[b]	0.209	1.421	0.0403	168.8	−0.87	2.45	
8322 Quartz[b]	0.175	2.989	0.1038	164.5	5.64	6.79	7.46
M-945 Feldspar[b]	0.194	1.348	0.0436	159.5	0.49	1.12	9.30
7108 Angostura Limestone[b]	0.188	1.542	0.0492	166.6	2.02		
C-8 Bridge Canyon Limestone[b]	0.202	1.870	0.0548	168.8	2.20		

[a] Brett Pit aggregate 77 per cent basalt.

[b] Thermal coefficient values were measured on the rock types shown. Other thermal properties values were computed from data obtained from concretes according to Section 6, Chapter I of Part VII, Boulder Canyon Final Report. In the case of basalt from the Brett Pit these other thermal properties were corrected for the 23 per cent other material present.

All aggregates are described in detail in Table I and the thermal properties values for both aggregate and concrete are given in Table II.

TEST RESULTS AND DISCUSSION

These tests were made to investigate the hypothesis that a differential in thermal coefficient of expansion between the fine and coarse aggregate or between the paste or mortar and the coarse aggregate will result in deterioration of the concrete containing such aggregates. Thus, if a paste with a thermal coefficient between 8 and 9 millionths[4] or a mortar with a thermal coefficient between 6 and 7 millionths were combined with an aggregate having a thermal coefficient between 1 and 2 millionths, the resulting concrete may be subjected to large internal stresses in climates where there are large variations in temperature. Smaller stresses may also be produced by differences in the diffusivity of concrete components.

It is not expected that these tests will yield quantitative results. The aggregates used were selected primarily because of their range in thermal properties. By the use of materials such as marble, quartz, feldspar, basalt, and limestone, an attempt was made to determine qualitatively whether variations in thermal properties could definitely be identified with deterioration in concrete. The discussion that follows points out that materials combinations that were expected to show deterioration in concrete did not always do so and that some combinations that were thermally compatible produced concrete that deteriorated quite rapidly in the various tests.

Weathering Tests:

Results shown in Figs. 2 and 3 are

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[4] Millionths of an inch per inch per deg Fahr.

from concretes made with marble, quartz, and feldspar, limestone, steel balls, and aggregate from Grand Coulee Dam. These concretes were subjected to one or more of the nine simulated weathering exposures shown in Fig. 1. In the tests represented by Fig. 2, only Exposure 2 or the 160 F variation (Exposure 3) of Exposure 1 produced serious expansion, while in the tests represented by Fig. 3 both Exposures 1 and 2 produced expansion. These expansions occurred in quartz, feldspar, combinations of quartz and feldspar, and in the Grand Coulee sand-feldspar gravel combination. The marble, limestone, and Grand Coulee aggregates produced only small expansions in concretes containing these materials.

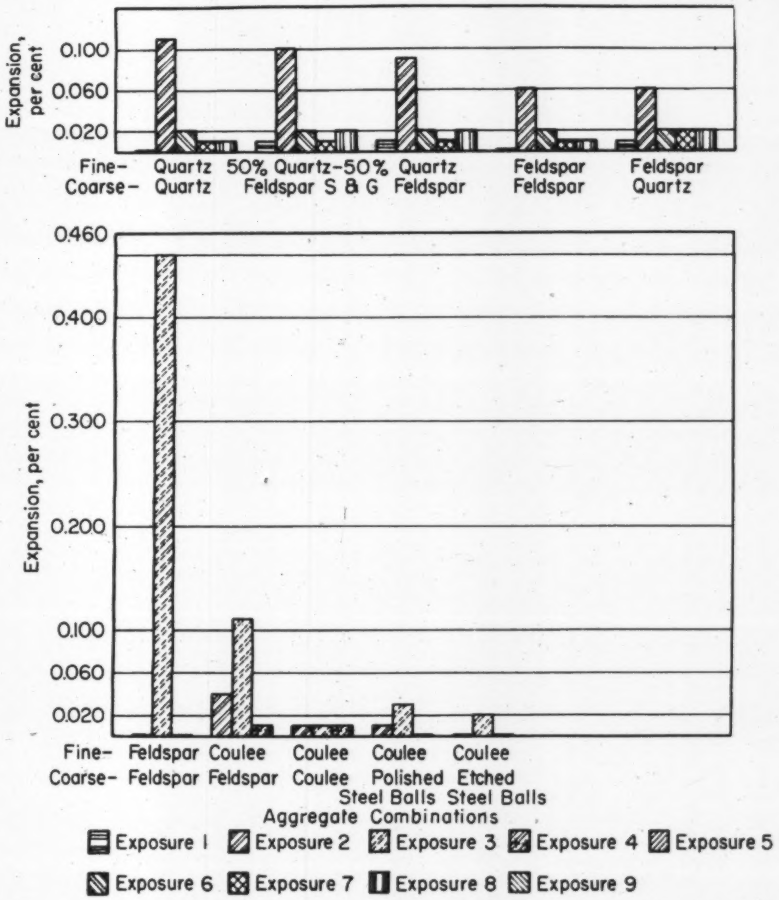

FIG. 2.—Expansion Caused by Simulated Weathering Exposures in Concretes Containing Various Types of Aggregates

The results of these tests do not confirm the hypothesis that prompted the investigation. Concrete containing marble aggregate gave very good results when subjected to the simulated weathering tests, yet marble has a low thermal coefficient of expansion and is anisotropic. Concrete made with limestone from Bridge Canyon Dam site also has a

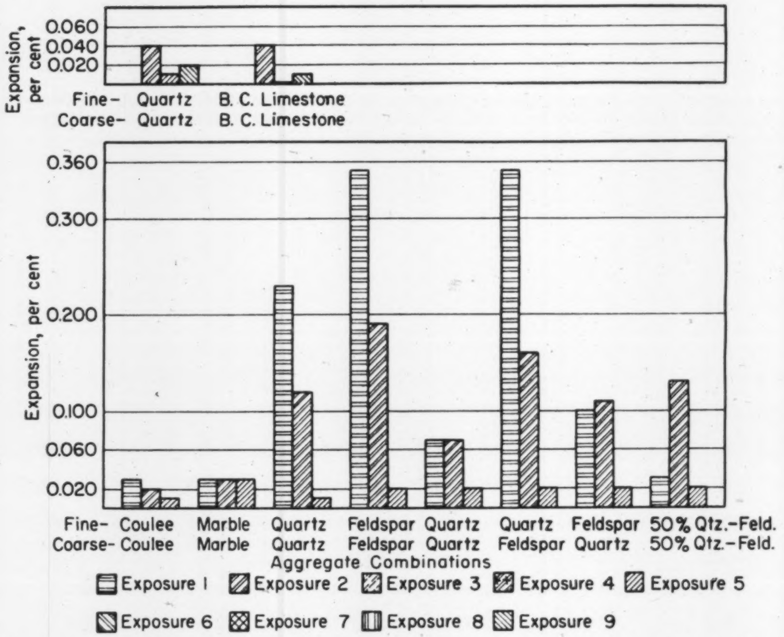

FIG. 3.—Expansion Caused by Simulated Weathering Exposures in Concretes Containing Various Types of Aggregates.

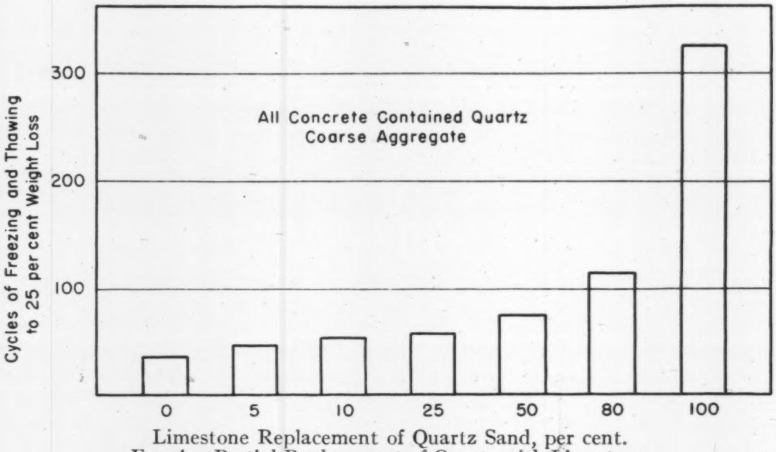

FIG. 4.—Partial Replacement of Quartz with Limestone.

relatively low thermal coefficient, yet it gave a high resistance in the simulated weathering tests.

Freezing and Thawing:

Concrete made with limestone from the Bridge Canyon Dam site gave low resistance in the freezing-and-thawing test, while concrete made with limestone from the Angostura Dam site gave high resistance although the thermal coefficients of both limestones are low. Quartz, which is slightly anisotropic but which had the highest thermal coefficient—nearest that of the mortar or cement paste—showed a very poor resistance to freezing and thawing and poor resistance to almost all the weathering tests to which it was subjected. Feldspar, which is highly anisotropic, gave poor resistance in all the tests as did all combinations of quartz and feldspar. These results indicate that differences in thermal coefficients of expansion do not play an important part in the deterioration of concretes.

Of the materials used in these tests, quartz, which has a relatively high thermal coefficient, should give good results when subjected to the temperature variations that occur in the accelerated freezing and thawing test. A partial replacement of quartz by limestone should provide an incompatible aggregate and should result in deterioration of a concrete made with the combination. Results shown in Fig. 4 are from concretes that contain combinations of limestone and quartz. These concretes all contain $\frac{3}{4}$-in. maximum size aggregate and the coarse aggregate in each concrete is crushed quartz. The bar marked 0 per cent contained all quartz sand and the one marked 100 contained all limestone sand, denoting that 100 per cent of the quartz sand in the all-quartz mix was replaced by limestone sand. Results of replacements of quartz sand by 5, 10, 25, 50, and 80 per cent limestone sand are shown in the other bars on Fig. 4. These data indicate that an increasing replacement of quartz by limestone in the sand sizes give increasing resistance to deterioration. This is contrary to what might be expected from a thermal incompatibility viewpoint.

Combined Tests—Heating-Cooling and Freezing-Thawing:

Accelerated freezing-and-thawing tests in air were made on concretes that had been subjected to 125 cycles of dry temperature alternations. Some of the specimens had been stored at 70 F, 50 per cent relative humidity; others had been alternated between 40 and 100 F, while others were alternated between −10 and 100 F. Length and weight change measurements were made on all specimens during the period of temperature alternations. The only differences noted were in the weights, which evidently changed because of moisture loss. The 40 to 100 F specimens lost most moisture, −10 to 100 F next, and 70 F storage the lost least moisture. The degree of cracking that subsequently occurred in the accelerated dry freezing-and-thawing tests is dependent on this moisture loss and, apparently, upon the aggregate used. These results are shown in Fig. 5. In these tests concrete containing quartz and the quartz sand-feldspar gravel showed the most serious cracking. Test of companion bar specimens in Exposures 1 and 5, after the temperature alternations, showed cracking in only the concretes containing Kimball and Republican River aggregates. These two materials were the only ones used in the tests that contained reactive materials. Examination of the bars showed evidence of alkali-aggregate reactivity; therefore the expansions of these bars were evidently

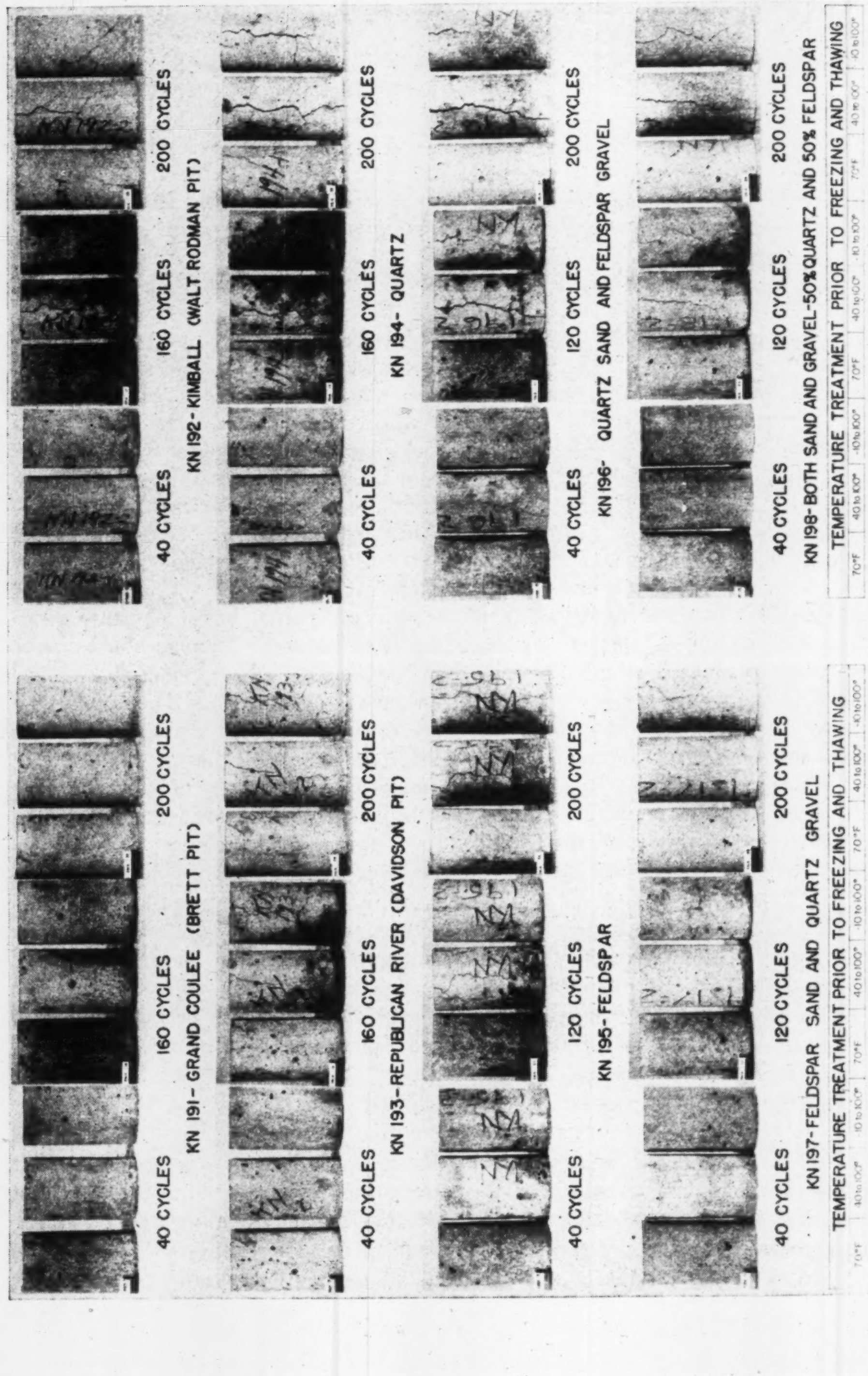
40 CYCLES
160 CYCLES
200 CYCLES
KN 191 - GRAND COULEE (BRETT PIT)
40 CYCLES
160 CYCLES
200 CYCLES
KN 193 - REPUBLICAN RIVER (DAVIDSON PIT)
40 CYCLES
120 CYCLES
200 CYCLES
KN 195 - FELDSPAR
40 CYCLES
120 CYCLES
200 CYCLES
KN 197 - FELDSPAR SAND AND QUARTZ GRAVEL
TEMPERATURE TREATMENT PRIOR TO FREEZING AND THAWING
70°F
40 to 100°
10 to 100°
70°F
40 to 100°
-10 to 100°
70°F
40 to 100°
-10 to 100°
40 CYCLES
160 CYCLES
200 CYCLES
KN 192 - KIMBALL (WALT RODMAN PIT)
40 CYCLES
160 CYCLES
200 CYCLES
KN 194 - QUARTZ
40 CYCLES
120 CYCLES
200 CYCLES
KN 196 - QUARTZ SAND AND FELDSPAR GRAVEL
40 CYCLES
120 CYCLES
200 CYCLES
KN 198 - BOTH SAND AND GRAVEL - 50% QUARTZ AND 50% FELDSPAR
TEMPERATURE TREATMENT PRIOR TO FREEZING AND THAWING
70°F
40 to 100°
-10 to 100°
70°F
40 to 100°
-10 to 100°
70°F
40 to 100°
-10 to 100°

FIG. 5.—Freezing-and-Thawing Results on High-Alkali Cement in Tests of Various Aggregates for Thermal Incompatibility.

due to this reaction. As in the results shown in Figs. 2 and 3, there is no evidence that thermal properties of either the aggregate or concretes contributed to deterioration.

Expansions Occurring During Thermal Properties Measurements:

While determining the thermal coefficient of saturated aggregates and concretes, it was noted that some specimens showed considerable expansion upon freezing. An example of this expansion is shown in Fig. 6. A saturated feldspar concrete decreased in length in the normal manner as the temperature was lowered to about 26 F. As the temperature continued to drop, the specimen showed tremendous expansion. A deviation at 15 F from the extended normal contraction line and the actual length change of the specimen is designated *D*. This specimen also showed a permanent set when returned to 60 F temperature and is designated as *SP* (Fig. 6). This curve was obtained by Mitchell (4) of the Bureau of Reclamation and is similar to those obtained by Valore (3) of the National Bureau of Standards in a slow freezing-and-thawing cycle.

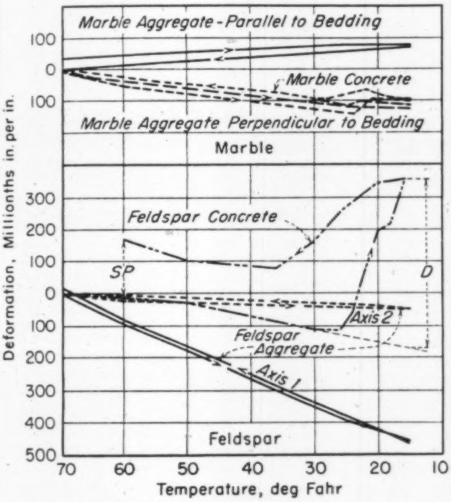

FIG. 6.—Length Change of Concrete and Concrete Aggregate During Freezing Cycle.

Although the saturated feldspar aggregate was thermally anisotropic, length change along the different axes remained straight lines. Thus, the aggregate was not disrupted by freezing action, but the saturated concrete containing the feldspar aggregate was.

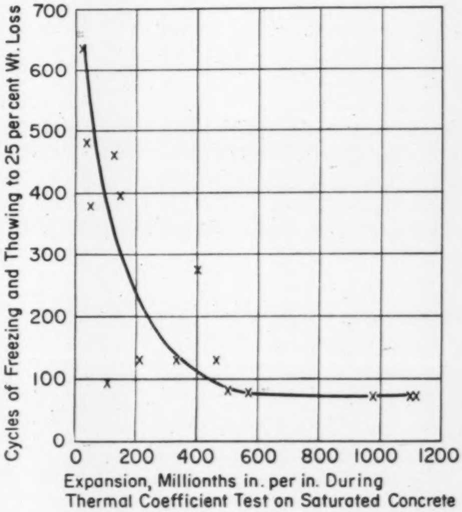

FIG. 7.—Expansion of Saturated Concrete During Measurement of Thermal Coefficient.

The marble specimen shown in this figure was also anisotropic and even had a negative coefficient along one axis. The saturated marble concrete exhibited only very slight expansion upon freezing.

Quartz aggregate showed slightly different coefficients along the three different axes, and length changes were normal in the freezing range. Quartz concrete, however, showed expansion very similar to the feldspar concrete. Limestone aggregate and concrete were similar to the marble; that is, showed no appreciable expansion on freezing.

Correlation of Expansion with Freeze-Thaw Durability:

A comparison of the accelerated freezing-and-thawing tests with the Expansion *D* (of Fig. 6) that occurs during the thermal properties test is shown in Fig. 7. The expansion measurements made during the thermal test were on (*a*) saturated concretes containing quartz, feldspar, marble; combinations of quartz, feldspar, and marble; two type of limestone, one from Bridge Canyon Dam site and the other from Angostura Dam site; on (*b*) the Bridge Canyon limestone combined with both quartz and local Cherry Creek sand; on (*c*) the Angostura limestone combined with natural sand from the Angostura Dam site; and (*d*) on the Bridge Canyon limestone sand combined with crushed quartz in the coarse-size fractions. Accelerated freezing-and-thawing tests were made on companion specimens that had been fog cured for 28 days prior to test.

From the results shown in Fig. 7, it seems probable that early failure of concretes can be predicted, especially when the measurement is made on saturated concrete. Data shown in Fig. 7 represent only a portion of the tests that have been made. Although some control was exercised over the degree of saturation in these tests, there was considerable variation in most of the specimens; consequently, correlation of the thermal test expansions with durability could not always be obtained. However, sufficient data, as shown in Fig. 7, have been obtained to indicate that a correlation will exist. Much more work must be done on the technique of the test, especially in controlling the degree of saturation, before a sure correlation can be developed and a quantitative evaluation made on the ability of various materials to produce durable concrete.

CONCLUSIONS

1. There is no uniform trend in this series of tests that indicates a direct dependency of durability on the thermal properties of the aggregates. Combinations of materials, such as marble coarse and quartz fine aggregate, which have high differences in thermal coefficients, showed good resistance to weathering action. Aggregates such as quartz and feldspar that have quite different diffusion constants gave uniformly poor results in concrete. Limestones with almost identical thermal coefficients gave quite different results in the freeze-thaw tests.

2. Data obtained during measurement of thermal coefficients of saturated concretes apparently provide a means for predicting the freezing-and-thawing durability of concrete containing various types of aggregates.

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DISCUSSION

MR. HUBERT WOODS.[1]—The *D* value reported is the expansion that takes place during the freezing part of the cycle used to determine the thermal coefficient of expansion. Therefore it is analogous to the expansion and in fact is the expansion that one can observe on a concrete undergoing one cycle of freezing if that concrete is in a saturated condition. Is it correct that from the expansion taking place in the first cycle you have a good idea of what is going to take place subsequently, and that the expansion is not necessarily related to the aggregate itself?

MR. ELMO C. HIGGINSON (*author*).—Not necessarily related to the thermal coefficient of the aggregate.

MR. BRYANT MATHER.[2]—The authors have emphasized the fact that there are many influences that control the durability of concrete and that, in the cases under discussion, factors other than those concerning the thermal coefficients of the component ingredients were those which controlled the durability of the concrete. I wish merely to emphasize the importance in my opinion of that realization, and I think that Mr. Scholer's recent remark in which he called attention to the importance of other properties of the ingredients fits into this same picture.

With the possible exception of the case described some years ago by Mr. Pearson,[3] there is little, if any, information to suggest that concrete has deteriorated from other than combined causes, and even in that case I suggest that there were a number of causes involved. The difficulty of selecting one cause and saying that it is primarily responsible for the deterioration of the concrete is a difficulty that must, I think, always be borne in mind when attempting to explain observed phenomena, either in the laboratory or in the field.

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[1] Director of Research, Portland Cement Assn., Chicago, Ill.

[2] Engineer, Concrete Research Division, Waterways Experiment Station, Corps of Engineers, Jackson, Miss.

[3] J. C. Pearson, "A Concrete Failure Attributed to Aggregate of Low Thermal Coefficient," *Journal*, Am. Concrete Inst., Vol. 38, p. 29 (1941).

THE SIGNIFICANCE OF TESTS FOR SULFATE RESISTANCE OF CONCRETE*

BY E. C. HIGGINSON[1] AND O. J. GLANTZ[1]

When water or soils containing sulfate salts of magnesium or sodium come in contact with concrete, a reaction takes place which is harmful to the concrete. Specifically, the sulfate salts react with the hydrated calcium aluminate of portland cement to form crystals of calcium sulfoaluminates which occupy a larger volume than the reactants. These crystals force the concrete to expand, thereby causing cracking to progress inwardly from the surface, which results in eventual disintegration.

Figures 1 and 2 show the formation of crystals of calcium sulfoaluminate and the resulting deterioration of the matrix in concrete which was exposed to sodium sulfate solution. Figure 1 is a photograph, having a magnification of 10×, of a 3 by 6-in. concrete cylinder which was exposed to a 2.1 per cent solution of sodium sulfate for one year in the laboratory. Figure 2 is a cross-sectional photograph of a piece of disintegrated concrete taken from a floor slab placed in contact with a soil containing a high concentration of sodium sulfate. Figures 3 and 4 show the results of sulfate attack on concrete floor slabs during an 11-yr period. The slabs were 20 ft wide, 120 ft long, and had a thickness of 4 to 5 in. The frame housing initially erected upon the floor slab was removed in 1947. Failure of the concrete appears to be the result of sulfate attack alone, since the concrete was placed in a warm, dry area, with nonreactive aggregates. Figure 3 is a general view; Fig. 4 shows the displacement, about 16 in., which occurred at an expansion joint as a result of the concrete expanding.

The problem of sulfate attack has long been recognized. Investigators have made field and laboratory studies (**1**, **2**)[2] of the phenomenon during the last 40 yr. For over 20 yr the Denver laboratories of the Bureau of Reclamation have been one of the groups performing research to find economical means of prevention. This paper summarizes test methods used by investigators and the results obtained to date.

TEST METHODS—GENERAL

Laboratory:

Test methods used in the various laboratories employ specimens of various sizes, shapes, and compositions. Small, thin, cement pats, rectangular bars ranging in size from 1 by 1 by 5 in. to 3 by 3 by 16¼ in., and cylinders 3 in. in diameter by 6 in. high, or 2 in. in diameter and 4 in. high have been successfully used. The choice of specimens in any investigation has been left to the judgment of the individual performing the test, except in the cooperative investigations conducted by the Working Committee on Sulfate Resistance of ASTM Committee

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Formerly, Head, Concrete Laboratory, and Materials Engineer, respectively, Bureau of Reclamation, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1010.

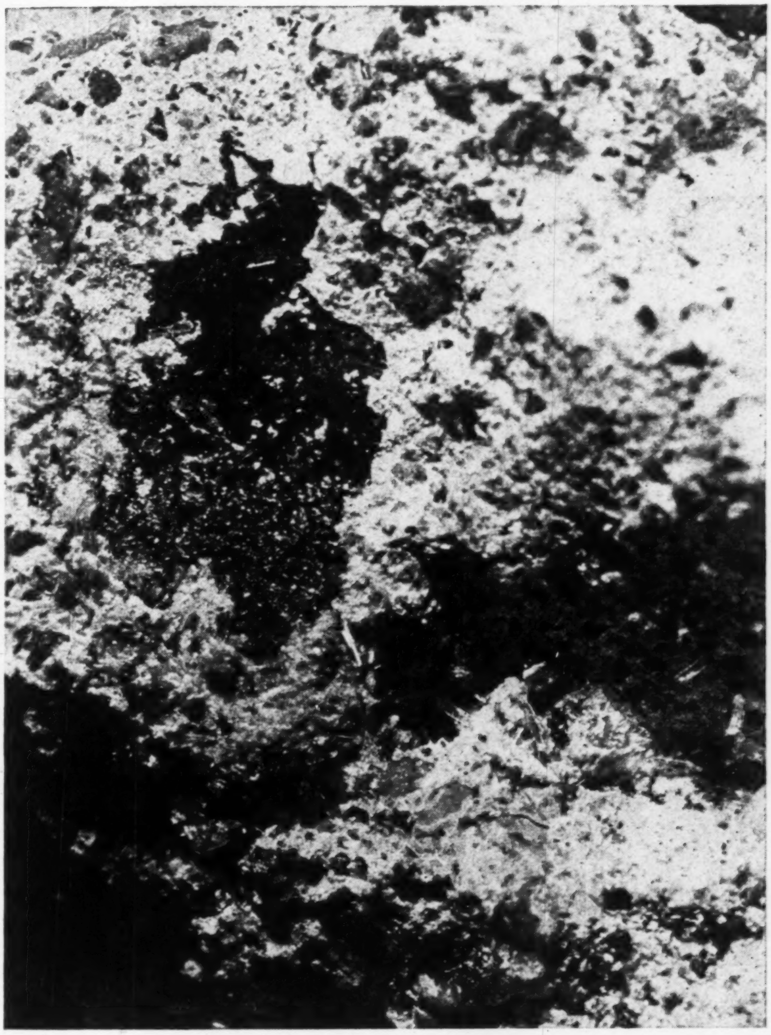

FIG. 1.—Formation of Crystals of Calcium Sulfoaluminates in Concrete Exposed to Sodium Sulfate Attack.

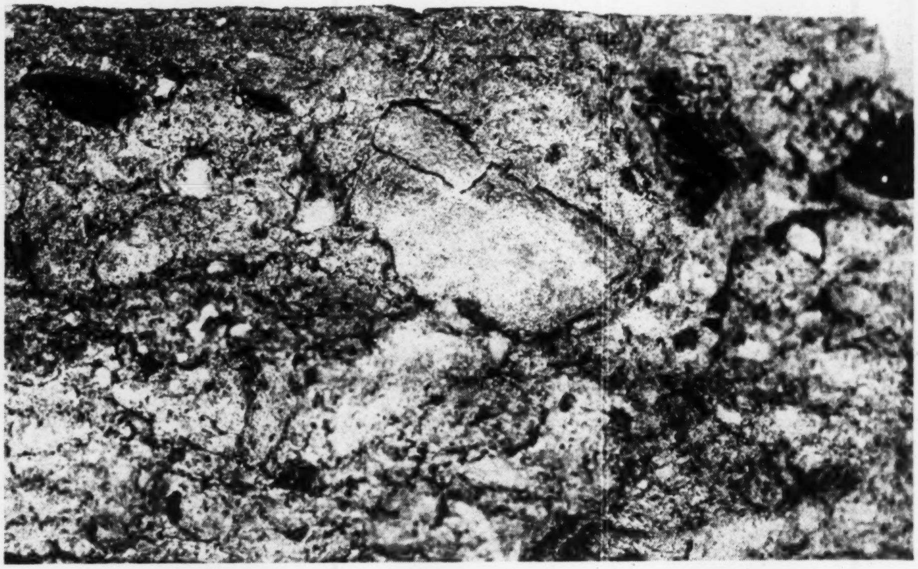

FIG. 2.—Concrete Disintegrated by Sodium Sulfate in Natural Soil.

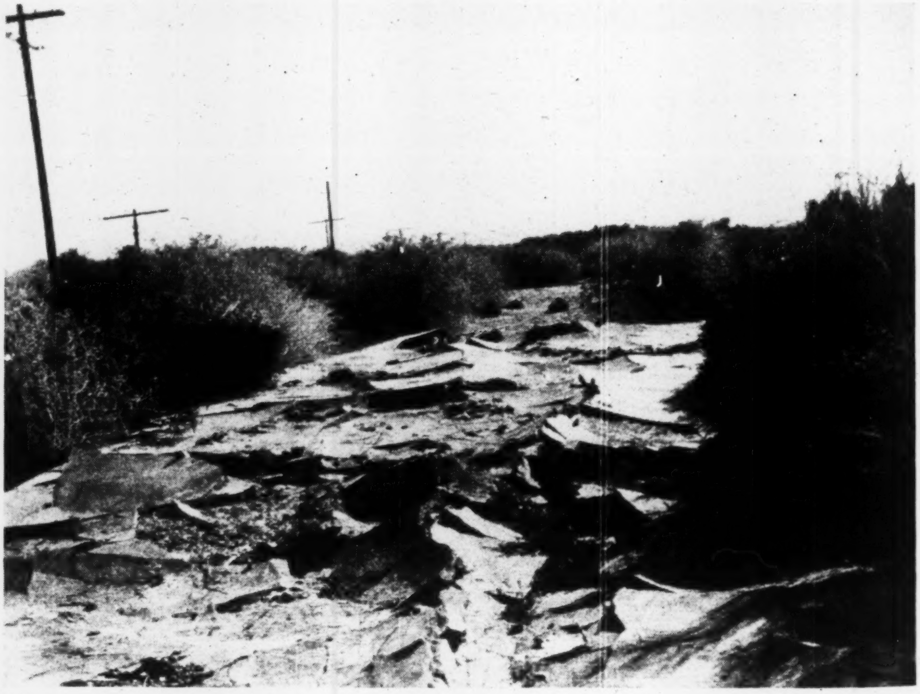

FIG. 3.—View of Disintegrated Concrete Slab (20 by 100 ft by 4 in. thick) by Sodium Sulfate Bearing Soil.

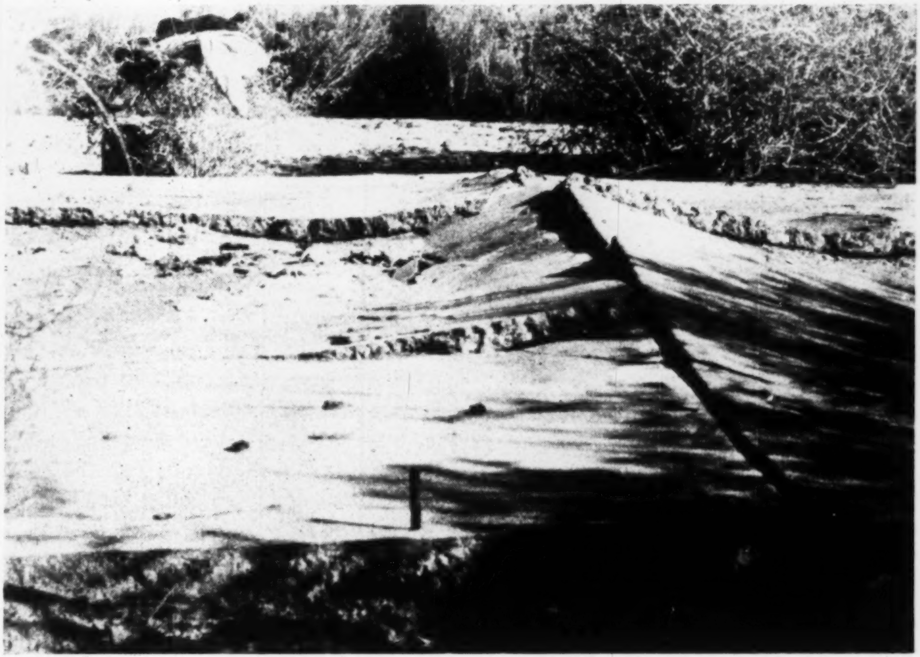

Fig. 4.—Displacement at an Expansion Joint, Resulting from Expansion of the Concrete.

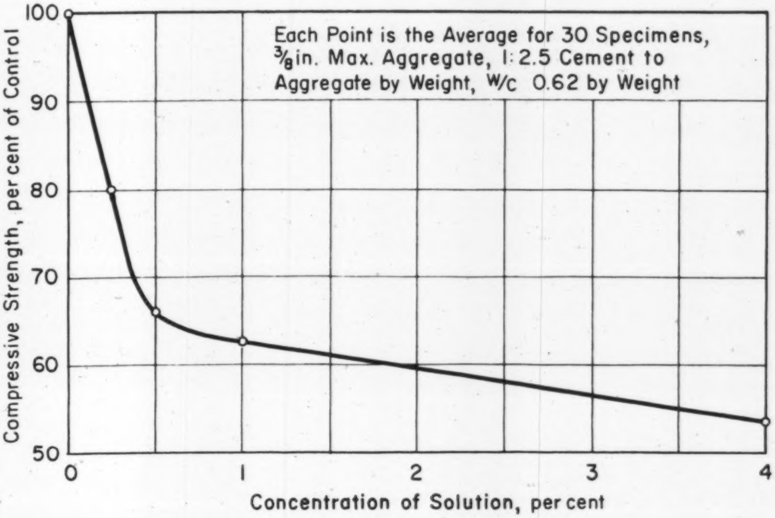

Fig. 5.—Effect of Concentration of Magnesium Sulfate Solutions on the Compressive Strength of Concrete Cylinders.

C-1 on Cement (3). In these tests, mortar consisting of one part cement to five parts Ottawa sand b· weight was cast into 1 by 1 by 10-in. bars.

Sodium sulfate solution, magnesium sulfate solution, or combinations of these solutions, in concentrations from 0.5 per cent to saturation have been used. Containers for holding the sulfate solution and the specimens are made of stainless steel, copper, ceramic crocks, or other material which is not corroded by the solution. The ratio of specimen volume to solution volume has varied over a wide range. Figure 5 shows the effect of concentration of magnesium sulfate solution on the compressive strength of concrete cylinders. This curve shows that increasing the concentration of the solution up to 0.5 per cent causes a marked decrease in strength as measured on specimens which have expanded 0.25 per cent. Increasing the concentration beyond 0.5 per cent has only a slight effect. Figure 6 shows Miller's (4) data on the variation of the rate of attack on concrete cylinders made with cements of varying tricalcium aluminate content stored in either magnesium or sodium sulfate solutions. It should be noted that the sodium sulfate solution is more harmful to concrete containing cements high in tricalcium aluminate (over 9 per cent) and that the magnesium sulfate solution is more harmful to concrete containing cements low in tricalcium aluminate (less than 8 per cent). Laboratory tests have shown that concrete exposed to solutions at temperatures between 40 and 100 F expands faster than concrete subjected to solutions at higher temperatures, with the fastest rate occurring at 100 F. Although this is an excellent temperature for obtaining fast laboratory test results, tests made at this temperature may be more expensive than those performed at lower temperatures.

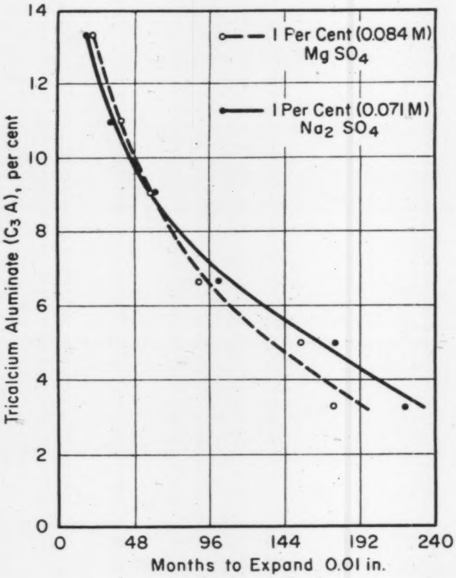

FIG. 6.—Effect of Tricalcium Aluminate Content of Cement on the Rate of Expansion of Concrete Exposed to Magnesium or Sodium Sulfate Solutions.

Testing procedures include innumerable methods of curing, test cycles, and criteria for determining the progress of sulfate attack. Both the methods of curing and the test cycles have included variations in the exposure period, the drying period, and the temperature at which drying is carried out. In addition to visual observation for determining the progress of attack, four other measurements have been used by various laboratories. These are changes in volume, weight, compressive strength, and modulus of elasticity as measured by the dynamic method.

Field:

Specimens used in field exposure tests have largely been 6 by 12-in. concrete cylinders and rectangular bars ranging from 3 by 3 by 15 in. to 6 by 6 by 48 in. Specimens have been exposed to sulfate-bearing soils, groundwater, and lakes containing large concentrations of sulfates. In a few instances, special exposure ponds have been constructed outdoors. Field exposure tests are subject to changes in climatic conditions, and in cold climates freezing and thawing makes evaluation of the sulfate attack more difficult. In general, field exposure tests are more difficult to evaluate than laboratory tests because they take place under uncontrolled conditions.

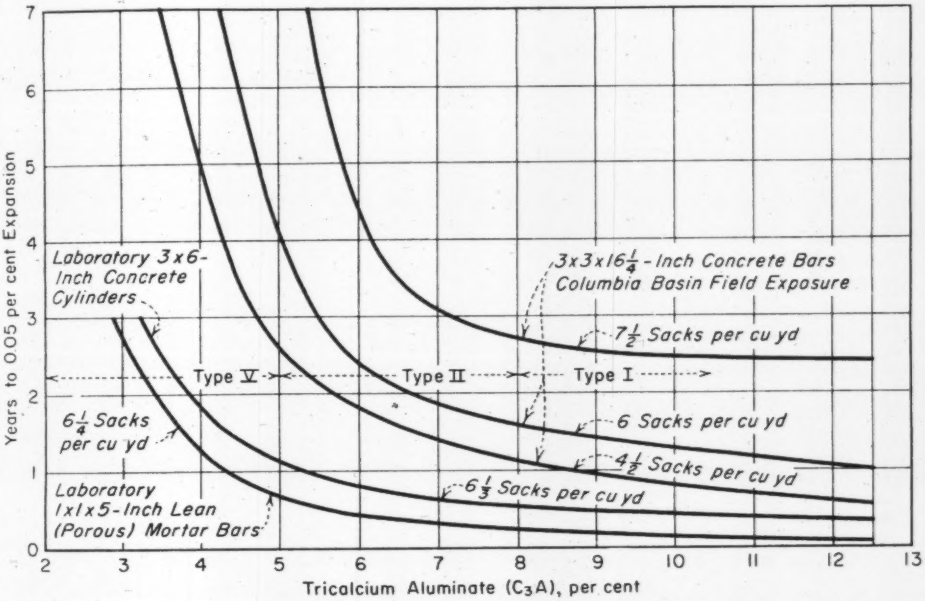

FIG. 7.—Effect of Tricalcium Aluminate Content of Cement and Richness of Mix on the Rate of Expansion of Concrete Exposed to Sulfate Waters.

BUREAU OF RECLAMATION TESTS

Laboratory:

Based upon the experience gained by several years of testing and many variations in specimens and procedures, a satisfactory test method has been developed by the Bureau of Reclamation. Standard specimens are 3 in. in diameter by 6 in. high and are fabricated from concrete containing aggregate graded up to $\frac{3}{4}$-in. maximum size; the grading being "standard" for all mixes. The water-cement ratio is fixed at 0.51 by weight, the slump at 3 in. and the air content at 5 ± 1 per cent. Cylinders are cured 14 days at 73.4 ± 3 F and 100 per cent relative humidity, followed by 14 days at the same temperature and 50 per cent relative humidity. The test cycle consists of 28 days immersion in 2.1 per cent sodium sulfate solution maintained at 73.4 ± 3 F, followed by 24 hr of drying in laboratory air.

The original length of each specimen is measured at the end of the 28-day curing period and just prior to immersion by a comparator accurate to 0.0001 in. Similar measurements are made at the end of each drying period and the expansion, based on the original length, is computed in per cent. When specimens have expanded 0.2 per cent, they are considered to have failed, and their age in days is recorded.

Field:

The Bureau has engaged in only one series of field tests to date. This investiga-

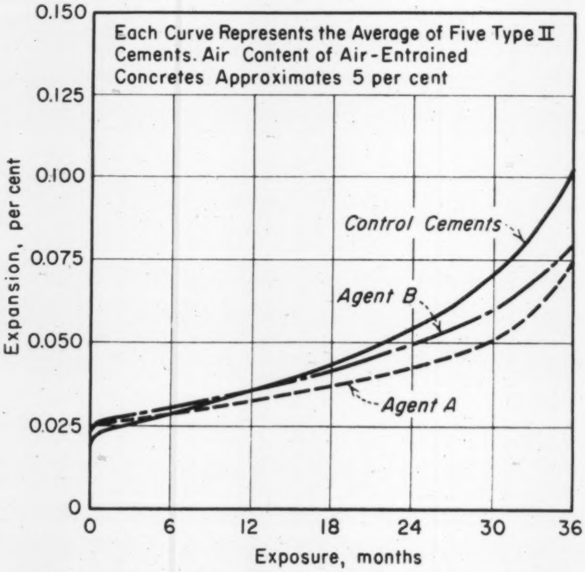

FIG. 8.—Effect of Air Entrainment of the Sulfate Resistance of Concrete.

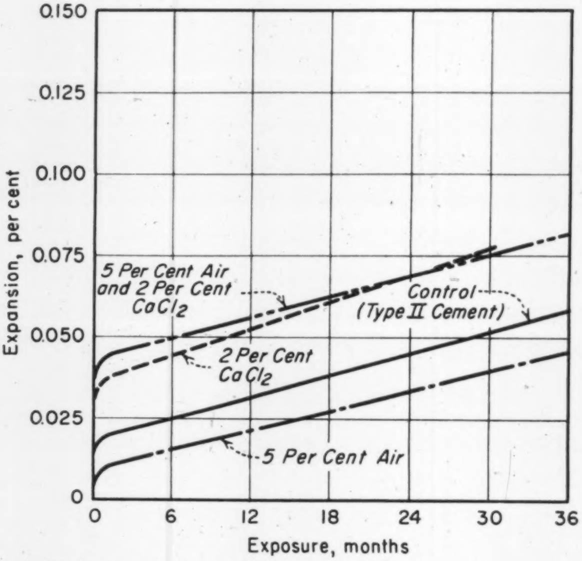

FIG. 9.—Effects of Calcium Chloride Additions and Air Entrainment on the Resistance of Concrete to Sulfate Attack.

tion was started in 1945 on the Columbia Basin Project in Washington and is still in progress. Eighteen cements, including ASTM types I, II, and V from seven plants located in the Northwest, are under test in concrete. Calcium chloride, one air-entraining agent, or both, were used in some specimens. A total of 96 different concrete mixes were made, from which 6 by 12-in. cylinders, 3 by 3 by $16\frac{1}{4}$-in. bars, and 6 by 6 by 48-in. posts were fabricated. The test specimens, with the exception of the large posts, are totally immersed in a 4 to 5 per cent solution of sulfate obtained by dissolving a natural salt cake that is predominantly sodium sulfate. The posts are only partially immersed so that the action at the water line might be observed. It is planned that this study will last over a period of fifty years. Concretes similar to those used in the field are also being tested in the Denver laboratories. The first analysis of the field and laboratory results was made at the end of six years, and for this analysis, the number of years required for the specimens to expand 0.05 per cent was selected as a basis for comparison.

The 6-yr test results are shown graphically in Figs. 7, 8, and 9. Figure 7 shows that the sulfate resistance is indirectly proportional to the tricalcium aluminate content of the cement in the concrete. Type V cement containing a maximum of 5 per cent tricalcium aluminate is most effective in protecting concrete subjected to severe attack. Increasing the cement content increases the sulfate resistance of concrete; however, $7\frac{1}{2}$ sacks of type II cement per cubic yard are required to make concrete approaching the same sulfate resistance as $4\frac{1}{2}$ sacks of type V cement. Figure 7 also shows that laboratory tests lead one to about the same conclusions as do field tests, but in a shorter period of time. Denver laboratory data shown in Fig. 8 indicate that air-entrainment does not materially affect the sulfate resistance of concrete. Five type II cements and two air-entraining agents were used: agent *A* a triethenolamine salt of a sulfonated hydrocarbon and agent *B* a calcium salt of a wood resin soluble in coal-tar solvent and insoluble in petroleum hydrocarbon solvent. In a few individual tests, air entrainment had a detrimental effect on the sulfate resistance. At equivalent air contents, there is some evidence that the agent used may affect the sulfate resistance slightly. A more comprehensive series of tests using several representative types of air-entraining agents is in progress.

Figure 9 shows that the addtion of 2 per cent calcium chloride to plain or air-entrained concrete has a deterimental effect on sulfate resistance. The magnitude of this effect is approximately the same when compared to the plain control concretes. Consequently, it appears that calcium chloride should not be added to either plain or air-entrained concrete which will be subjected to sulfate attack.

Pozzolanic Material:

The effects of pozzolanic materials on the sulfate resistance of concrete have not been fully determined. Completed preliminary tests and those now in progress indicate that the use of any of the various types of pozzolanic materials should not be expected to increase or decrease significantly the resistance of concrete to sulfate attack. There are some data, however, that indicate the use of either water-cooled slag or fly ash slightly improves this resistance while calcined diatomaceous earths, clays, and shales have the opposite effect, and in approximately the same magnitude. The paucity of completed tests, performed under present test conditions, precludes a detailed discussion of this phase of sulfate resistance.

CONCLUSIONS AND RECOMMENDATIONS

Tests on sulfate resistance of concrete have contributed greatly to the useful knowledge in the field of concrete technology. Some of the more significant conclusions and recommendations which may be made as a result of the tests follow:

1. Cements low in tricalcium aluminate content should be used in concrete structures which may be in contact with sulfate-bearing soil or water. If the concentrations are 0.1 to 0.2 per cent water soluble sulfates in the soil or 150 to 1,000 ppm sulfates in the water, type II cement with a tricalcium aluminate content of 8 per cent or less may be used. If, however, the sulfate content of the soil is over 0.2 per cent or the water contains over 1,000 ppm sulfates, the use of a type V cement, which by definition has a tricalcium aluminate content of 5.0 per cent or less, is recommended (5).

2. Increasing the cement content of concrete increases its sulfate resistance.

3. Entraining air in concrete in general increases its resistance to sulfate attack.

4. Calcium chloride reduces the resistance of concrete to sulfate attack.

5. Standardization of laboratory test procedures for sulfate resistance would be a worth-while advancement.

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(5) U. S. Bureau of Reclamation, *Concrete Manual*, Tables 2 and 5.

DISCUSSION

MR. T. R. GALLOWAY.[1]—It is frequently stated that pozzolanic material has been beneficial in avoiding the so-called alkali-aggregate reaction and in increasing sulfate resistance.

We have been trying to determine why the reactions of fly ash when used as a substitute for some of the portland cement in concrete are not entirely predictable. It seems that the variability may tie in with the amount of lime formed when the concrete sets.

We find very little in the literature with regard to the amounts of lime that appear in straight portland-cement concrete when it sets and with regard to what happens to that lime as the concrete ages. We have, therefore, examined specimens of straight portland-cement concrete and of fly ash concrete in which 20 per cent of fly ash was substituted for cement. These specimens were taken on recent jobs and the oldest of those examined were 210 days old. We have also analyzed some specimens taken from buildings several years old. In general, chemical and petrographic methods were used. In part of our work a few checks were attempted by X-ray diffraction. In the recent specimens of straight portland-cement concrete we found calcium hydroxide with a little calcium carbonate. In the old specimens we found calcium carbonate with a little calcium hydroxide. In the fly ash concrete specimens we found calcium hydroxide in amounts varying with the age of the specimens.

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[1] Structural Engineer, Consolidated Edison Company of New York, New York, N. Y.

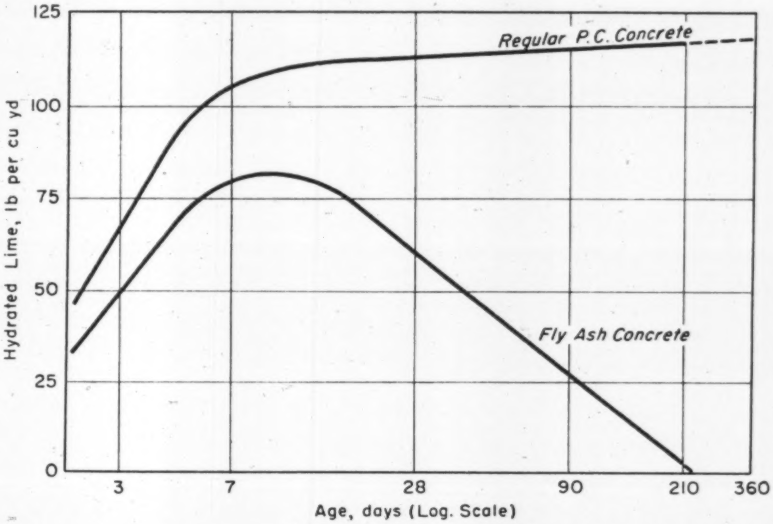

FIG. 10.—Fly Ash Activity. Reduction in Hydrated Lime in Hardened Job Concrete Due to Waterside Fly Ash.

The average result of 64 tests for hydrated lime in hardened regular portland-cement concrete was 115 lb per cu yd, which is equivalent to 18 lb per bag of portland cement.

The abscissas of the accompanying Fig. 10 represent age in days and the ordinates per cent of lime (reported as hydroxide). The upper curve is for straight portland-cement concrete and shows about 3 per cent of lime in all specimens after about 5 days. Assuming that a cubic yard of concrete weighs 4000 lb there are about 115 lb of hydrated lime per cu yd present after setting. The lower curve is for 20 per cent fly ash concrete and shows increasing amounts of hydrated lime (but less than in straight portland-cement concrete) up to about five days, with the amount of hydrated lime then decreasing until it becomes nearly zero at 210 days. During this period the strength of the fly ash concrete increased and at about 90 days surpassed that of the straight cement concrete.

It is well known that pozzolanic materials react with hydrated lime, producing some of the same compounds as are present in cement. This action accounts for the increase in strength of fly ash concrete over straight cement concrete. We suspect that the elimination of hydrated lime may account for the general improvement in the other properties of fly ash concrete over straight portland-cement concrete. In particular the improvement in the so-called alkali-aggregate reaction may be accounted for if hydrated lime is also an accessory to this reaction and the improvement in sulfate resistance if the reaction of the sulfate is with hydrated lime to form a compound similar to a double salt with calcium aluminate, rather than a reaction with calcium aluminate alone.

MR. D. O. WOOLF[2] (*by letter*).—If for no other reason, publication of this paper is warranted by the inclusion of Figs. 3 and 4. The condition of the concrete shown in these figures is almost beyond belief. The fact that this destruction of concrete is due to attack by common salts of the earth causes the reader to consider with much more thought the installation of concrete structures at locations where sulfates are prevalent.

One subject not covered by the paper but which is quite suitable for inclusion is the matter of identification of concrete which has failed by the action of sulfates. The authors state that the formation of crystals of calcium sulfoaluminate is the cause of the destruction of concrete exposed to sulfate salts. However, this chemical, calcium sulfoaluminate, will be found in almost all concrete to a greater or lesser extent and is described by many authorities as the result of a normal reaction between the gypsum and tricalcium aluminate. A distinction between the sulfoaluminate formed from gypsum and that formed from sulfates would be very desirable. It is assumed that in the case of attack by sulfates, a very considerable development of calcium sulfoaluminate would result. But deposits described as "heavy" of the sulfoaluminate have been stated to occur in concrete which has not been subjected to attack by sulfates. Consequently, a statement of the amount of the deposits which could be considered to be the result of the reaction involving gypsum would be helpful. If it is not feasible to describe or illustrate failed concrete showing crystals of calcium sulfoaluminate which were not developed from attack by sulfates, would it be possible to identify concrete which has been attacked by sulfates by the presence of crystals of the sulfate instead of the sulfoaluminate?

MR. ARTHUR F. PILLSBURY[3] (*by letter*).—The authors are to be congratulated upon supplying, in brief, easily read form, additional information on the sulfate resistance of concrete. I have no comment upon the paper itself, but would like to call attention to one thing. Many engineers, in deciding upon the need for sulfate-resistant concrete, fail to interpret correctly the effects of the hydraulic gradients of the moisture in the concrete and contacting soil. (The hydraulic heads are usually negative.) These gradients affect the concentration of the salts in the soil water or pore water, thus the need for sulfate resistance of the concrete. As an illustration, with a concrete pavement, there will normally be an upward gradient through the soil and concrete to the surface, where evaporation and the consequent concentration of salts can take place. Sulfate resistance is required if there are sufficient sulfates in the soil and if rainfall is too infrequent to keep salts flushed off the surface and to provide leaching through temporary gradient reversals. On the other hand, with any water pipe line, where the hydraulic head in the pipe is above the water table in the soil (a normal condition, except with drain tile), the gradient will be outward through the pipe walls. The concrete will be affected by the sulfates in the pipe water, not by those in the soil water (steel shell pipe excluded).

MR. W. C. HANNA.[4]—My own experience has been that calcined diatomaceous earths, pumice and calcined Monterey shale have in every case tended to make the cement mortars more resistant to sodium sulfate attack.

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[2] Physical Research Engineer, Bureau of Public Roads, Washington, D. C.

[3] Professor of Irrigation, College of Agriculture, University of California, Los Angeles, Calif.

[4] Vice President in Charge of Technical Development, California Portland Cement Co., Colton, Calif.

The laboratory test procedure that we have followed for many years as a means of studying sulfate resistance is as follows: The two ends of briquets, after making the customary 28 day tensile tests, are used as our test specimens. The ends of the briquets are dried with a towel, weighed and placed in solutions of sodium sulfate. Three are placed in a 2 per cent solution and three in a 10 per cent solution. Each set of three halfs represent the three briquets. Covered beakers are used for storage. The specimens are examined at regular intervals and any changes noted. After each examination, the solutions are changed.

In the 10 per cent sodium sulfate solution, all specimens start to show the effect of sulfate water at early ages, and in the 2 per cent solution a little later. Good resistance to sulfate action by this test is indicated when there is only a slight disintegration at 18 months in the 2 per cent sulfate solution.

A typical set of test results is given below to show that with 25 per cent of calcined Monterey shale, substituted for a like amount of portland cement, the resistance to sodium sulfate solution is greater than with the straight portland cement.

Type II Portland Cement (No. 23062)

Time	2 per cent Na_2SO_4	10 per cent Na_2SO_4
14 days	No action	Slight action
2 months	Slight action	....
4 months	....	All bad
18 months	Fair condition, gain in weight of 3.1 per cent	Gain in weight of 10.7 per cent

Type II P.C. with 25 per cent of fine Monterey Shale (No. 23065)[a]

Time	2 per cent Na_2SO_4	10 per cent Na_2SO_4
1 month	....	Slight action
3 months	Slight action	....
5 months	....	All bad
18 months	Fair condition, gain in weight of 0.7 per cent	Gain in weight of 3.7 per cent

[a] The type II cement used in making up the blend was part of Sample No. 23062.

From the appearance and the gain in weight noted at 18 months, the sulfate resistance of the cement with Monterey shale is decidedly greater than the cement without the shale.

We have also noted that field concrete with Monterey shale when exposed to sea water has indicated even better results.

Messrs. Raymond E. Davis[5] **and Milos Polivka**[6] (*by letter*).—No quantitative evidence is given in the paper in support of the statement made with respect to calcined diatomaceous earths, clays and shales appearing to be detrimental as judged by the results of laboratory tests and by the condition of field structures—it is so contrary to our findings of the past twenty years as to warrant discussion.

Concrete in service is subjected to a variety of conditions of sulfate exposure, none of which can be exactly duplicated in the laboratory any more than can conditions of freezing and thawing. Many structures such as pipe, drain tile, canal linings, etc., are subject to drying very infrequently if at all. Others, such as retaining walls, footings, and slabs resting upon or partially buried in the ground, may have surfaces which are exposed to long cycles of drying. And still others, such as piles, piers, etc., in ocean water, are subject to more or less regular and frequent cycles of wetting and drying. Regardless of the conditions of exposure, the alkali sulfates in solution which penetrate concrete react with the calcium hydroxide and calcium aluminate hydrate present in the hardened cement paste to form calcium sulfate (gypsum) and calcium sulfoaluminate. As these compounds crystallize from solution, expansive forces are set up within the capillaries which tend to disrupt the concrete. The stronger the alkali-sulfate solution, the more permeable and weaker the concrete; and the larger the quantities of calcium hydroxide and hydrated calcium aluminate present in the hardened cement paste, the more rapidly will deterioration occur.

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[5] Consulting Engineer and Director Emeritus, Engineering Materials Laboratory, University of California, Berkeley, Calif.

[6] Assistant Professor of Civil Engineering, University of California, Berkeley, Calif.

Under the first of the above-mentioned conditions, where there is little or no drying action, the solution penetrates all surfaces of the concrete in substantially like degree and like concentration. If in sufficiently strong solution, the alkali sulfates combine chemically with calcium hydroxide and hydrated calcium aluminate to form crystals of the reaction products in the capillaries at and near the exposed surfaces, temporarily decreasing the permeability of a thin outer layer of concrete. As these crystals grow and expansive forces of sufficient magnitude are developed, distintegration of this outer layer occurs, and a deeper layer is subject to similar attack. And so the process of disintegration continues.

Under the second of the above-mentioned conditions, where some of the surfaces of the structure are exposed to sulfate waters and others are exposed to drying, the apparent effect of chemical attack may take quite different forms. If the concentration of alkali sulfates is low, as is more usually the case, there is no significant deterioration at the wetted surfaces but due to evaporation from the dry surfaces and capillary flow towards these surfaces, there develops a gradually increasing concentration of the reaction products (calcium sulfate and calcium sulfoaluminate) not at the exposed surfaces but at some distance within the concrete mass. This causes an internal growth which is usually accompanied by severe cracking at the exposed surfaces and often by the splitting away or spalling of portions of sound concrete due to the large expansion and perhaps disintegration of concrete below.

Regardless of the type of exposure it seems evident that for optimum resistance to sulfate action the concrete should be as impermeable as practicable and the hardened cement paste should be low in the reactive compounds, calcium hydroxide, and hydrated calcium aluminate.

The paper does not state the conditions of test upon which the authors based their statement that "calcined diatomaceous earths, clays, and shales appear to be detrimental." We assume that this statement may have been based on the results of tests conducted in accordance with their preferred method as given in the paper. If so, the test specimens were 3 by 6-in. concrete cylinders, standard-cured for 14 days and then dried in an atmosphere of 50 per cent relative humidity for 14 days; and the testing cycle consisted of 28-day immersion in a 2.1 per cent sodium-sulfate solution, followed by 24 hr of air drying. These conditions can hardly be considered as representative of those which would normally obtain for any concrete structure in actual service. The 14-day drying of the small cylinders after only 14 days of curing would have a deleterious effect, not only on ultimate strength but also on watertightness of the concrete to a much greater degree than would be the case for the thicker and more massive concrete of field structures from which the percentage of moisture lost by evaporation would be relatively small. It is to be expected that this deleterious effect on strength and watertightness would be more pronounced for concretes which gain their strength slowly than for those which gain their strength rapidly.

The effect of pozzolanic action is well known. A suitable pozzolan used in appropriate amount in the presence of moisture will in time combine with the calcium hydroxide that is liberated during the process of hydration of portland cement to form calcium-silicate hydrate, which is essentially insoluble and immune to sulfate attack, which significantly contributes to strength of concrete, and which, by filling the capillaries greatly contributes to watertightness.

For the average portland cement, perhaps 75 per cent of the calcium hydroxide is liberated during the first seven days of hydration. During this early period of hydration, the pozzolan, depending upon its character and fineness, combines with the calcium hydroxide at a rate less rapid than that at which the calcium hydroxide is formed. For the less rapidly reactive pozzolanic materials, it is likely to be several months before the stage is reached when all of the then available calcium hydroxide has been combined, and thereafter the calcium hydroxide which is liberated by further hydration of the cement reacts with the pozzolan as soon as it is formed. Hence, during the early period of hardening, concretes containing pozzolan in the normal amounts of cement replacement may be expected to be not only of lower strength but also more permeable to the passage of moisture than corresponding concretes containing straight portland cement; however, at the age of 6 months the permeability of the concrete containing a suitable pozzolan in appropriate amount is generally only a small fraction of that of the straight portland-cement concrete.

The authors' method of drying small specimens between the ages of 14 and 28 days and then saturating them with sulfate solution as the cyclic tests are begun would naturally place the concretes containing pozzolanic materials at a great disadvantage as compared with concretes containing normal portland cements. This disadvantage would not obtain for most field structures with their less severe conditions of exposure and more massive section which, even if partially exposed to the air, would retain much of the original moisture until the pozzolanic reaction, strength, and watertightness had been more completely developed.

These are all matters to which we have given considerable thought and attention over the years, and our studies have led us to the opinion that the results of laboratory tests made on specimens continuously immersed in sulfate solutions are the best index to the probable sulfate resistance of field structures in general. For specimens thus treated, three criteria may be employed in judging the resistance of mortars and concrete to sulfate action, as follows:

1. Disintegration as measured by loss in weight,
2. Decrease in compressive strength as compared with that of corresponding concrete of the same age continually immersed in water, and
3. Volume change or increase in length as compared with that of corresponding specimens immersed in water.

For a wide variety of cement compositions and a wide variety of pozzolanic materials used as cement replacements, there is generally good correlation between loss in weight, decrease in compressive strength, and increase in length.

By any one of these criteria we have found that calcined pozzolans of suitable composition, employed in appropriate amounts, greatly increase the resistance to sulfate action of mortars and concretes containing type I cements of high tricalcium aluminate content, substantially increase the resistance of mortars and concretes containing type II cement of moderately low tricalcium aluminate content, and to some extent increase the resistance of mortars and concretes containing type V cement. In support of this statement we have extracted data from publications originating in the Engineering Materials Laboratory of the University of California and from private reports with which the writers have been concerned. These data are presented in the following tables.

The values of the accompanying Table I[7] show the relative resistance of concretes containing certain fly ashes, used as a cement replacement in the amount of 20 per cent by weight, as compared with corresponding concrete containing straight type II portland cement. As judged by compressive strength at the age of 6 months, after 5-month immersion in sodium-sulfate solution the deleterious effect of sulfate action was substantially greater for the straight portland-cement concrete than for any of the fly-ash concretes. For the Chicago fly ash, with its very low carbon content, the compressive strength of treated specimens was actually higher than that of the untreated specimens, though the difference would be within the probable experimental error. Even for the less desirable fly ashes with 12 or 19 per cent carbon, sulfate resistance, as judged by decrease in compressive strength, was in the order of 30 to 50 per cent greater than that for the type II cement alone.

TABLE I.—SODIUM SULFATE RESISTANCE OF CONCRETES CONTAINING FLY ASH.

1:5.6 concrete by weight; ¾ in. maximum aggregate; Type II cement; composition: C_3S = 50 per cent, C_2S = 25 per cent, C_3A = 6 per cent, C_4AF = 15 per cent.

Fly Ash in Portland-Pozzolan Cement			Percentage of Fly-Ash Replacement, by weight	$\frac{W}{C + F}$, by weight	Compressive Strength, Age 6 Months, psi		Change in Compressive Strength, due to Treatment, per cent[b]
Type	Carbon, per cent	Specific Surface, sq cm per g			Standard-Cured Specimens	Treated Specimens[a]	
None	....	....	0	0.43	6310	5560	−12
Chicago	1.1	3220	20	0.40	6950	7120	+2
West Penn	6.4	3080	20	0.43	6800	6580	−3
Long Island	10.4	3800	20	0.46	6650	6400	−4
Potomac	11.9	2390	20	0.45	6420	5830	−9
Hell Gate	19.3	1970	20	0.49	5480	5060	−8

[a] 3 by 6-in. specimens standard-cured for 28 days, then immersed in 10 per cent solution of sodium sulfate. Tested at age of 6 months.

[b] Change in compressive strength between treated and standard-cured specimens.

Table II gives the results of tests in which two calcined opaline shales were employed as replacements in the amounts of 25 and 35 per cent of portland cement, with and without air-entraining or water-reducing agents. It will be noted that the cement contained less than 5 per cent tricalcium aluminate (C_3A) and only 9 per cent tetracalcium aluminoferrite (C_4AF). For all practical purposes it met the ASTM requirements for a sulfate-resistant cement.

Phillips shale comes from a deposit of the Monterey formation high in opal near Pomona, Calif. owned by the California Portland Cement Co. This shale was employed in the construction of Davis Dam. Napa earth is a cream-colored shale, high in opal and perhaps of diatomaceous origin, though only an occasional diatom structure can be identified. It occurs in a deposit in Napa County, Calif. owned by the Basalt Rock Co., and was used by the East Bay Municipal Utility District in all pipe and structures of the recently completed East Bay outfall-sewer project.

The values of Table II show that without air-entraining or water-reducing admixture, the effect of immersion in 10 per cent sulfate solution from the age of 28 days to 1 yr was to reduce the compressive strength of straight portland-cement concrete by 14 per cent. When Phillips shale was employed in the amount of 25 per cent, the treated specimens exhibited a compressive strength

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[7] The data were taken from R. E. Davis, R. W. Carlson, J. W. Kelly, and H. E. Davis, "Properties of Cements and Concretes Containing Fly Ash," *Proceedings*, Am. Concrete Inst., Vol. 33, pp. 577–612 (1937).

2 per cent greater than that of the untreated specimens. The use of admixture C, which entrained no air, generally decreased sulfate resistance. The other three admixtures, all of which entrained air, increased sulfate resistance. With admixture A and Phillips shale in the amounts of 25 and 35 per cent, sulfate action up to the age of 1 yr had no effect upon compressive strength of concrete, as compared with 8 per cent reduction in compressive strength of the corresponding concrete without Phillips shale. With agent C, which entrained no air, and Phillips shale in the amount of 35 per cent or Napa earth in the amount of 25 per cent, there was only a 2 per cent reduction in strength due to sulfate action, as compared with a 23 per cent reduction in strength of the straight portland cement with agent C only. Further study of the table shows that either Phillips shale or Napa earth, with or without air-entraining or water-reducing admixture, contributed very significantly to sulfate resistance when used with portland cement of composition considered to be highly resistant to sulfate action.

TABLE II.—SODIUM SULFATE RESISTANCE OF CONCRETES CONTAINING PHILLIPS SHALE AND NAPA EARTH.

1:6.7 concrete by weight; ¾-in. maximum size aggregate. Cement composition: C_3S = 54.3 per cent; C_2S = 27.1 per cent; C_3A = 4.7 per cent; C_4AF = 8.8 per cent.

Calcined Pozzolan[a]		Admixture[b]	$\frac{W}{C+P}$, by weight	Slump, in.	Air Content,[c] per cent	Compressive Strength, Age 12 months, psi		Change in Compressive Strength, per cent
Type	Percentage by weight of (C + P)					Standard-Cured Specimen	Treated Specimen[d]	
None	..	None	0.52	3.7	3.2	5600	4840	−14
Phillips shale	25		0.61	3.7	2.3	4970	5060	+2
None	..	A	0.49	3.6	5.7	5400	4950	−8
Phillips shale	25		0.57	3.7	5.7	5200	5200	0
Phillips shale	35		0.58	3.7	5.7	4890	4890	0
Napa earth	25		0.59	4.0	5.7	4970	4820	−3
None	..	B	0.48	3.8	5.7	5140	4800	−7
Phillips shale	25		0.56	3.7	5.7	5090	4890	−4
None	..	C	0.48	3.7	3.5	6340	4880	−23
Phillips shale	25		0.53	3.6	2.6	6960	6020	−14
Phillips shale	35		0.54	3.6	2.8	5990	5850	−2
Napa Earth	25		0.55	3.7	3.2	6760	6620	−2
None	..	D	0.44	3.7	4.8	6150	5440	−12
Phillips shale	25		0.49	3.7	4.4	6390	6370	0
Napa earth	25		0.50	3.9	5.1	5830	5770	−1

[a] Phillips shale from California Portland Cement Co. deposit near Pomona, California, calcined at about 1500 F and ground to a Blaine fineness of 13,000 sq cm per g. Napa earth from Basalt Rock Co. deposit in Napa County, Calif., calcined at 1650 F and ground to a Blaine fineness of 22,000 sq cm per g.

[b] Admixtures A, B, D are air-entraining agents; C is a water-reducing agent.

[c] Air content by pressure method.

[d] Sulfate resistance determined by immersing eight 3 by 6-in. cylinders in 10 per cent solution of sodium sulfate at age 28 days. A standardized air jet used once a month to remove loosened material. At age 12 months, four cylinders tested for reduction in compressive strength in comparison with standard-cured specimens.

The data of Table II were taken from a private report to the East Bay Municipal Utility District, dated March, 1949, and are here submitted for publication with the permission of the District.

The values of Table III were drawn from the results of a rather extensive test program in which a variety of pozzolanic materials was employed.[8] In this cooperative program, the Metropolitan Water District of Southern California, under the direction of Mr. L. H. Tuthill, performed the tests to determine sulfate resistance.

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[8] Reported in part in R. E. Davis, J. W. Kelly, G. E. Troxell, and H. E. Davis, "Properties of Mortars and Concretes Containing Portland-Pozzolan Cements," *Proceedings*, Am. Concrete Inst., Vol. 32, Sept.-Oct., 1935, pp. 80–114.

Type I cement was employed, for which the tricalcium-aluminate content was 11 per cent. The pozzolans were used as replacements for portland cement in the amount of 20 per cent by weight. The source of each pozzolan is given in the footnote to the table. All of the pozzolans were calcined. Two are classed as opaline shales, two are clays, and two are volcanic glasses. Concrete specimens were completely immersed in 1 and 10 per cent solutions of sodium sulfate.

From an examination of the table, it will be seen that the concrete containing straight portland cement was completely disintegrated in the 10 per cent solution at 5.2 months and was completely disintegrated in the 1 per cent solution at 1 yr. The concrete containing clay A immersed in the 10 per cent solution completely disintegrated in 3 months, and that immersed in the 1 per cent solution completely disintegrated in 10.5 months. However, the use of clay C greatly improved sulfate resistance as judged by the compressive strength of concrete specimens immersed in the 1 per cent sodium sulfate solution up to the age of 1 yr, and also in the 10 per cent sodium solution up to the age of 6 months. Of particular note is the performance of the concrete in which was employed Monterey shale calcined at 1800 F. Immersion for a period of 6 months in the 10 per cent sodium-sulfate solution produced no deleterious effect upon strength.

Table IV shows the effect of raw and

TABLE III.—SODIUM SULFATE RESISTANCE OF PORTLAND-POZZOLAN CEMENT CONCRETES.

1:5.7 concrete by weight; ¾-in. maximum size aggregate. Type I cement, composition: C_3S = 59 per cent; C_2S = 22 per cent; C_3A = 11 per cent; C_4AF = 6 per cent.

Group	Pozzolan[a]	Temperature of Calcination, deg Fahr	$\frac{W}{C+P}$, by weight	Compressive Strength,[b] psi				Change in Compressive Strength,[c] per cent Sodium Sulfate Solution		Avg. Monthly Change in Compressive Strength,[c] per cent 10% Sodium Sulfate Solution, Age 6 months
				Water Curing		Sodium Sulfate Solution				
				6 months	1 yr	1 per cent, 1 yr	10 per cent, 6 months	1 per cent, 1 yr.	10 per cent, 6 months	
Portland Cement	None	..	0.56	4860	5160	12 mo.[d]	5.2 mo.[d]	−100.0	−100.0	−19
Opaline Shales	Diatomaceous shale[e]	1450	0.60	3620	3890	3830	2760	−1.5	−23.8	−4
	Monterey shale[f]	1450	0.68	4680	4920	4850	3950	−1.4	−15.5	−3
	Monterey shale[f]	1800	0.60	5490	5580	5040	5580	−9.7	+1.6	0
Volcanic Glasses	Pumicite[g]	1450	0.58	5650	5340	6060	4540	+13.5	−19.5	−3
	Tuff	1450	0.58	5120	5350	4920	4700	−8.0	−8.2	−1
Clays	Clay A[i]	1450	0.58	4200	4350	10.5 mo.[d]	3 mo.[d]	−100.0	−100.0	−33
	Clay C[j]	1450	0.56	4580	5030	4760	3410	−5.4	−25.5	−4

[a] 20 per cent by weight of portland-pozzolan cement.

[b] 3 by 6-in. cylinders; standard-cured 28 days, then immersed in water or in sulfate solution as indicated.

[c] Change in compressive strength between treated and water-cured specimens.

[d] Complete disintegration at age indicated.

[e] Naturally oil-impregnated low-grade diatomaceous shale from Airox Co. deposit near Casmalia, Calif.

[f] A shale of the Monterey formation from Santa Cruz Portland Cement Co. deposit near Davenport, Calif.

[g] Pumicite from deposit near Friant, Calif.

[h] From Monolith Cement Co. deposit near Monolith, Calif.

[i] From Santa Cruz Portland Cement Co. deposit near Davenport, Calif.

[j] Known as Fleming Clay, from California Portland Cement Co., Colton, Calif.

calcined diatomaceous earth upon the resistance to sulfate action, as judged by loss in weight of concrete completely immersed in 10 per cent solutions of both sodium sulfate and magnesium sulfate. The diatomaceous earth was obtained from the Johns-Manville deposit at Lompoc, Calif. Its silica content was about 80 per cent, and its fusion temperature was about 2450 F. Type I cement was employed, for which the C_3A content was 13 per cent.

TABLE IV.—SODIUM AND MAGNESIUM SULFATE RESISTANCE OF CONCRETES CONTAINING DIATOMACEOUS EARTH.

1:6.7 concrete by weight; ¾-in. maximum size aggregate. Type 1 cement; composition: $C_3S = 48$ per cent; $C_2S = 26$ per cent; $C_3A = 13$ per cent; $C_4AF = 7$ per cent.

Diatomaceous Earth			$\frac{W}{C + P}$, by weight	Slump, in.	Flow, per cent	Compressive Strength,[a] psi, 30 months	Period of Continuous Immersion at 70 F to 25 per cent Loss in Weight[b]			
							10 per cent Sodium Sulfate		10 per cent Magnesium Sulfate	
Type	Specific Surface, sq cm per g	Percentage by Solid Volume of $(C + P)$					Months	Relative per cent	Months	Relative per cent
None	..	..	0.53	2.8	73	5940	5	100	9	100
Raw	21 000	12	0.57	2.7	73	5690	7	140	15	167
Calcined at 2340 F	7 000	12	0.53	2.9	73	6660	16	320	18	200
Calcined at 2340 F	7 000	24	0.54	2.9	74	6310	24+[c]	1000[e]	24	267
Calcined at 2450 F	6 000	24	0.53	2.8	74	6080	24+[d]	1090[e]	24	267

[a] Standard curing for 18 months, then in air at 70 F and 95 per cent R.H. until 3 weeks prior to test, then in water at 70 F until test.

[b] 3 by 6-in. cylinders standard-cured 28 days, then immersed continuously at 70 F in 10 per cent solution of sodium sulfate or magnesium sulfate equal to volume of specimens; solution replaced every 28 days.

[c] 12 per cent loss at 24 months.

[d] 11 per cent loss at 24 months.

[e] Computed on basis of average monthly loss in weight.

The data given in the table have been extracted from a private report, entitled "Tests of Mortars and Concretes Containing Diatomite," made to Johns-Manville Corp. in 1948. They are here submitted for publication with the permission of Johns-Manville.

A study of the table will show that, judged by 25 per cent loss in weight, the straight portland-cement concrete failed in 5 months when immersed in the sodium-sulfate solution and in 9 months when immersed in the magnesium-sulfate solution. The use of raw diatomite as a substitution for portland cement in the amount of 12 per cent by absolute volume considerably improved resistance to each of the sulfates. Calcination at 2340 F (below the temperature of incipient fusion) led to further improvement. Doubling the amount of calcined diatomaceous earth to 24 per cent by absolute volume led to a very great increase in sulfate resistance. For concretes containing this larger percentage of replacement, the loss in weight was only 11 or 12 per cent after 2 yr of immersion in the sodium sulfate solution (as compared with 25 per cent loss in weight after 5 months immersion for the straight portland-cement concrete); in the magnesium sulfate solution, failure was judged to have taken place in 24 months (as compared with 9 months for the concrete containing straight portland cement).

From these results, it is quite evident that the resistance to sodium sulfate action of concrete containing an appropriate amount of calcined diatomite of the quality here under consideration is many times as great as that of corresponding concrete containing a typical type I cement; and the resistance to magnesium-sulfate action of concrete containing an appropriate amount of calcined diatomaceous earth is several times as great as that of the corresponding straight portland-cement concrete.

In conclusion, it is our opinion that, in light of the overwhelming evidence to the contrary, it would be most unfortunate if the statement of the paper concerning the deleterious effect of calcined diatomaceous earths, clays, and shales should come to be regarded as valid for the better pozzolanas in these groups and for the conditions of curing and exposure to which most field structures will be subjected.

Additional references on this general subject are given below.

Lewis H. Tuthill, "Resistance of Cement to the Corosive Action of Sodium Sulfate Solutions," *Proceedings*, Am. Concrete Inst., Vol. 33, Nov.–Dec., 1936, pp. 83–106.

R. E. Davis, "Pozzolanic Materials and Their Use in Concretes," Symposium on Use of Pozzolanic Materials in Mortars and Concretes, Am. Soc. Testing Mats., p. 3 (1949). (Issued as separate publication *ASTM STP No. 99.*)

H. S. Meissner, "Pozzolans Used in Mass Concrete," Symposium on Use of Pozzolanic Materials in Mortars and Concretes, Am. Soc. Testing Mats., p. 16 (1949). (Issued as separate publication *ASTM STP No. 99.*)

R. E. Davis, W. C. Hanna, and E. H. Brown, "Strength, Volume Change, and Sulfate Resistance of Mortars Containing Portland-Pozzolan Cements," Symposium on Use of Pozzolanic Materials in Mortars and Concretes, Am. Soc. Testing Mats., p. 131 (1949). (Issued as separate publication *ASTM STP No. 99.*)

WEAR TESTS OF CONCRETE*

By H. L. Kennedy[1] and M. E. Prior[1]

Synopsis

A brief history of early tests used to evaluate the resistance of road materials and concrete to abrasion is presented.

The types of wear generally experienced on concrete surfaces are broken down into four general categories and the causes discussed in each case.

Methods of test used by investigators to evaluate the wear resistance of concrete are discussed for the various types of wear. The advantages and disadvantages of the test procedures have been considered and comments made regarding the proper type of test for a particular kind of wear.

The effect of such factors as strength and curing of the concrete have been included in the discussion, since they have a very important bearing on the ability of the concrete to resist wear.

Seldom is there any agreement as to what wear actually is or how to measure it. Some feel that the old paint slogan, "Save the surface and you save all," is applicable to concrete. Certainly, when the surface is disrupted and the aggregate exposed, it is difficult to measure the abrasion resistance of the resulting concrete surface. This is particularly true when means are adopted—and some with merit—to increase the life of the wearing surface through the use of various concrete surface hardeners.

It has been demonstrated that cement factor, air content, and curing are all important factors. But all three factors follow the compressive strength of concrete, so that it may be reasonably hypothesized that strength may be accepted as a criterion of wear resistance.

It would appear that a reasonable solution to the problem is possible and an acceptable method of test may be devised, if only concrete and the factors that go into making and curing that concrete are considered. It appears equally obvious that concretes treated for improving their resistance to wear involve a different procedure from that used when the concrete only is the prime factor.

As an example, a very inferior concrete may be painted with a rubber-base paint and the resistance to the action of the shot-blast machine is extremely high until the surface is penetrated. When this surface is penetrated, the resistance to abrasion so measured is ridiculously slight by comparison. In other words, the actual strength of the concrete is then the controlling factor.

The first work which seems to have been reported on wear tests of paving materials was by Johann Bauschinger

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Manager, Construction Specialties Division, and Head, Construction Specialties Research Laboratory, respectively, Dewey and Almy Chemical Co., Cambridge, Mass.

(1)[2] of Munich in 1844. He used a machine, first exhibited at the World's Fair in Paris in 1878, similar to the Dorry machine, in which cores are subjected to abrasion by silica sand carried on a revolving cast steel plate. This work, and much of the early work in this country as reported by Page (2) and Goldbeck and Jackson (3), was conducted on rock and stone then used for paving.

In more recent years, adaptations of these and other methods for determining the abrasion resistance of stone have been used with varying degrees of success on concrete. The investigations of Roman (4), Abrams (5), Jackson and Pauls (6), Scofield (7), and Scholer and Allen (8) were with the rattler-type equipment, such as the Deval test and the Los Angeles and Talbot-Jones rattlers. About the same time, Guttman (9) reported on a disk type of test. A drill press adaptation was also reported by Harris (10) in connection with the testing of the hardness of various materials. Tuthill and Blanks (11) classified concrete wear into two general types, mechanical and hydraulic. They state that no one test has been devised that adequately measures the wear resistance of concrete under all conditions.

There are a number of various types of abrasion or wear to which concrete may be subjected. Webster's dictionary defines "wear" in this way—"To impair, waste or diminish by continual attrition, scraping, percussion or the like." The problem seems to be to discover or to select from existing equipment one kind that could be generally accepted for determining the abrasion resistance of all concrete.

It will not be possible, within the scope of this paper, to cover all tests, but those believed applicable to present-day concrete will be considered.

TYPES OF ABRASION

Wear of concrete surface by abrasion will be classified as follows for the purpose of this paper:

1. Wear on concrete floors, due to foot traffic, light trucking, and the skidding or sliding of objects on the surface (attrition).
2. Wear on concrete road surfaces due to heavy trucking and automobiles, with and without chains (attrition plus scraping plus percussion).
3. Wear on underwater construction, due to the action of abrasive materials carried by flowing or turbulent waters (attrition plus scraping).
4. Wear on dams, power plants, and water-carrying systems where a high hydraulic gradient is present. This is generally known as cavitation erosion as distinguished from the abrasive erosion in type 3 (percussion).

The first type of wear listed is essentially a rubbing action and is usually caused by the introduction of foreign particles, such as sand, metal scraps, or similar materials. Normal wear without the benefit of such abrasive materials would be negligible on a good concrete surface for an indefinite period of time. A poorly designed or inadequately cured surface would, of course, wear down readily.

The second type of wear is caused by a rubbing action similar to that found in the first type, plus an impact-cutting type of wear. This latter type is brought about by the use of chains on automobile and truck tires or metal vehicle wheels. As the wheel revolves, it brings the metal into contact with the concrete surface with considerable impact, a process which tends to cut the surface of the concrete. Here again, mix design, finishing, and curing are important factors in the ability of the concrete to resist this action.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1031.

The third type of wear is primarily a cutting action. The action of the abrasive particles carried by the flowing water is, of course, controlled largely by the velocity of the water, the angle of contact, the abrasive material, and the general surrounding conditions.

The fourth type of wear is completely an impact abrasion. It is caused by the abrupt change in direction and velocity of a liquid to such a degree that the pressure at some point is reduced to the vapor pressure of the liquid. The vapor pockets so created upon entering areas of higher pressure collapse with a great impact, which eventually causes pits or holes in the concrete surface. Also, the particles torn loose by this action continue to add to the abrasion problem by causing further wear as designated by the third type previously mentioned. Wallace and Price (**12, 13**) have reported on the Bureau of Reclamation's extensive studies of this problem in connection with various dams. Since this is a highly specialized problem, it will not be covered further in this paper.

It will be realized, from the foregoing, that probably no one single type of abrasion test can be considered adequate. The rubbing type may be satisfactory for floor surfaces, but in no way does it simulate the cutting action of sand carried by fast-moving water. The dressing-wheel type does approach the cutting action produced by chains, but its action is somewhat dependent upon the hardness of the coarse aggregate used in the mix. This is, of course, also true in the case of the rubbing type. The sand-blast type cuts into the softer materials, leaving the harder materials exposed or loosened so that they are free to become dislodged from the mass. This type of action is, however, more severe than that commonly found in floors; it cuts through the surface finish and exposes the basic concrete which, under normal conditions of wear, might not be exposed during the life of the building.

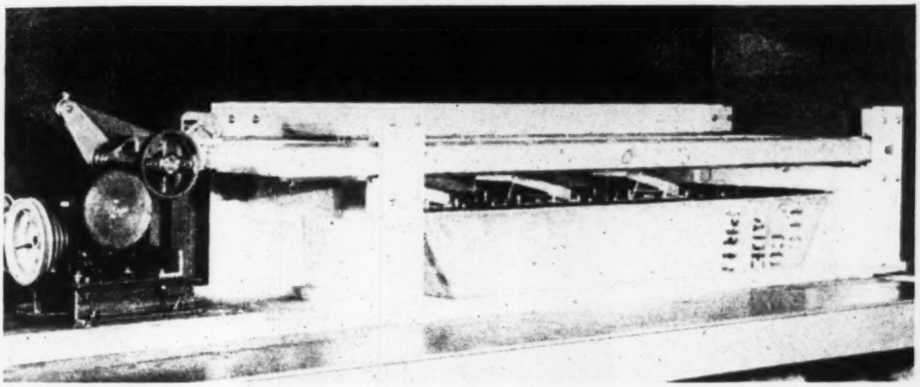


FIG. 1.—Reciprocating Shoe Abrasion Device Developed by Research Laboratories, Lehigh Portland Cement Co.

TYPES OF SURFACES

Floor Surfaces:

A considerable amount of work has been done by many investigators in connection with the study of the wearing qualities of concrete floors. Kessler (**14**), Ahlers *et al.* (**15**), Shank (**16**), Emley and Hofer (**17**), Wastlund and Eriksson (**18**), and Schuman and Tucker (**19**) have reported on the developments of test methods and results of investigations. This work has involved various finishing techniques, types of curing, time of curing, degree of curing, integral floor hardeners, surface hardeners, and air entrainment. Also included in these studies were the effects of paints, acids, and alkalies on the resultant surfaces in respect to the ability of the surface so treated to resist abrasion.

Most of these studies have been carried out by rubbing types of apparatus, since this was considered to be the method best suited to reproduce the actual action on the floor surface. The other two types, dressing-wheel and sand-blast, have also been used, but to a limited extent.

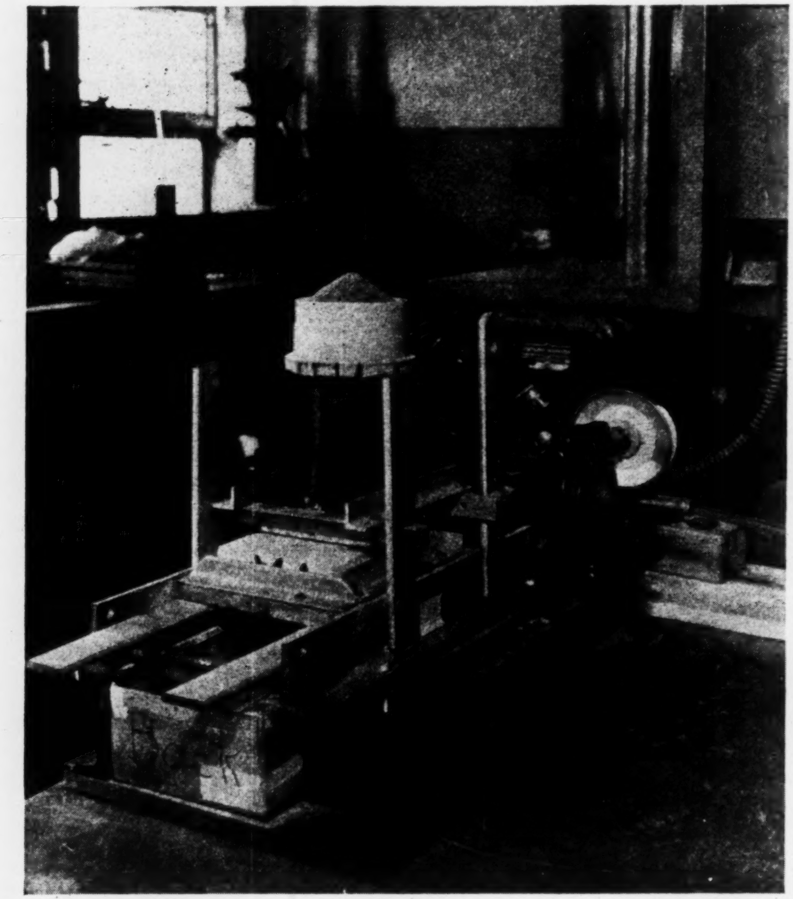


FIG. 2.—Reciprocating Abrasive Machine Developed by Research Laboratories, Public Service Gas and Electric Co.

The two common methods of achieving this rubbing action are a reciprocating disk and a revolving disk with some sort of abrasive material—usually carborundum, silica sand, or slag, used under damp or wet conditions. The length of time required to obtain significant results depends mainly on the abrasive material used, the pressure applied, and the speed of operation, provided the surface characteristics of the specimens under test are comparable. This time may vary from 2 to 20 hr.

Figures 1 and 2 illustrate two of the reciprocating types of machines currently being used in wear test studies.

The revolving disk-type machine commonly used is essentially that developed by Schuman and Tucker (**19**). Variations of this apparatus have been constructed by laboratories interested in studying the wear resistance of floors, and reasonable results have been obtained.

There is always the question of the effect of the aggregate particles, particularly where the surface had no special treatment such as the use of integral or surface hardeners. Under these conditions, the surface finish wears off quickly and the abrasive disk will then proceed to ride on the hardest piece of aggregate. In actual practice, however, the traffic

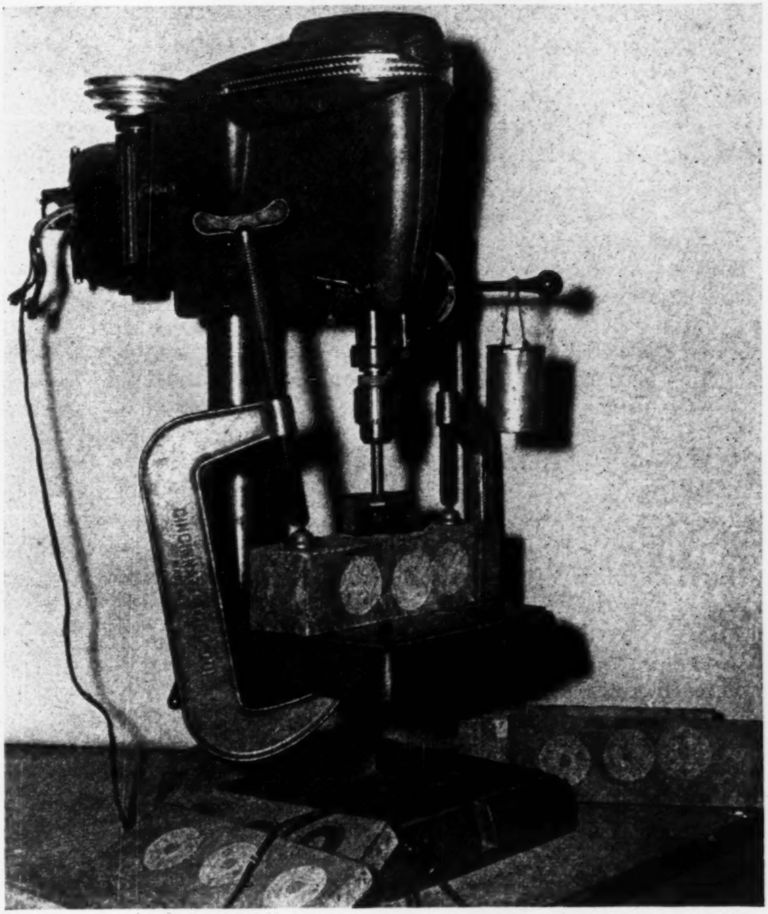


FIG. 3.—Typical Drill Press Set Up for Dressing Wheel Type of Testing. Used by Northeastern University.

will wear around the harder particles, leaving them protruding and susceptible to impact.

In general, where the conditions of test are such that only a surface hardness is to be examined, the rubbing action will produce satisfactory results. This includes toppings containing an integral hardener, surface hardeners, various types of finishing, and the application of film-forming materials such as paints.

The dressing-wheel type of test, Fig. 3, has also been found to be suitable for this sort of test. It is, in general, much more rapid in action than the rubbing type and is a fairly simple piece of equipment. It can be set up in a drill press and does not require an abrasive or water. General practice is to clean the surface occasionally during the test by blowing the dust off the specimen under test.

When the wear caused by the wheel has progressed through the surface of the concrete, there is again the tendency for the hardest aggregate particle or particles to carry the burden. However, this condition is not so pronounced with the wheel as with the rubbing test, because some of the teeth will be making contact at other points. There will be a tendency for the wheel to bounce because of high and low spots, thus causing gouging. The extent of this bouncing will be controlled to a great degree by the loading pressure applied to the wheel and the speed of operation. The use of an abrasive and lower loading pressures tends to overcome this undesirable action.

The shot-blast test, Fig. 4, may be successfully used for surface testing, provided due care is exercised to control the rate of abrasion. It is possible to adjust the rate of flow of shot so that within a reasonable period of time it will affect only the surface; thus the surface treatment can be evaluated. On the other hand, the length of test or the flow of shot can be increased to provide a more severe test that will cut around the aggregate and loosen it, regardless of its relative hardness.

The use of paints, particularly the rubber-base type, changes the effectiveness of the various tests. In the case of the shot blast, there is a tendency for the shot to bounce off the surface without cutting because of the resiliency of the film. Thus, this test becomes of no value. The shot blast, of course, will cut through a water-base paint or similar film without difficulty. However, both the rubbing action and the dressing wheel will wear through the paint film to the surface of the concrete, and it is possible to get a measure of the effectiveness of the paint as a protective coating.

Floor hardeners of the surface-applied type also pose a problem because they do not generally provide a very thick surface-hardened condition. The more mild form of abrasion obtained by the rubbing action is more sensitive to slight differences in surface hardness than the other types of test methods. However, it is doubtful if these slight differences are of actual value for practical purposes.

Floors that are subjected to acid conditions, such as in milk plants and citrus fruit packing plants, very often become damaged by the acid action to such an extent that the abrasion resistance is considerably lowered. This reduction in resistance can be measured by all three methods under discussion. However, in cases of this type, it is the mortar that is weakened and, therefore, the shot blast is by far the most effective method. The dressing wheel would rank second and the rubbing action a poor third. The trouble with the rubbing action is that it will, as previously stated, tend to ride on the aggregate, thus greatly masking the true condition of the concrete.

Roads:

The investigation of the abrasion resistance of roads dates back to the earliest tests that were actually applied to paving stone. Since the introduction of concrete, there have been a few published reports concerning the abrasion resistance of concrete roads. Collins and Waters (20) and Wastlund and Eriksson (21) have published reports on abrasion tests of highway concrete. This work is in a somewhat different category from

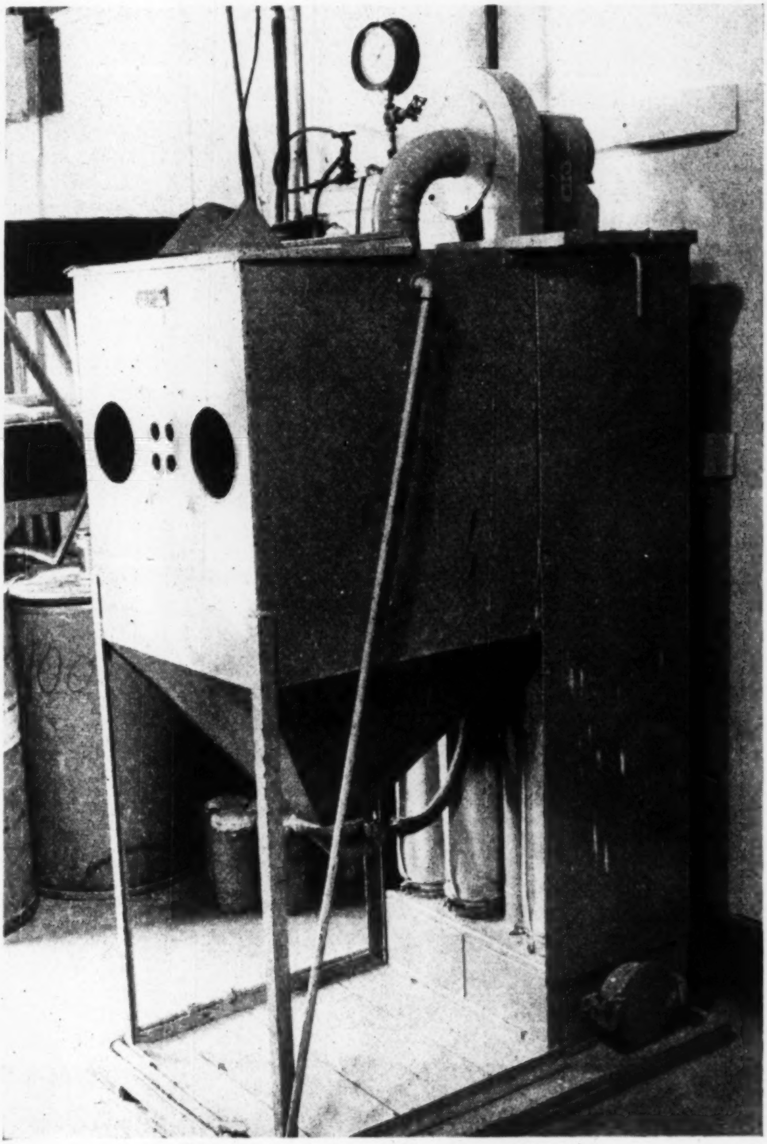


FIG. 4.—Typical Shot-Blast Test Cabinet.

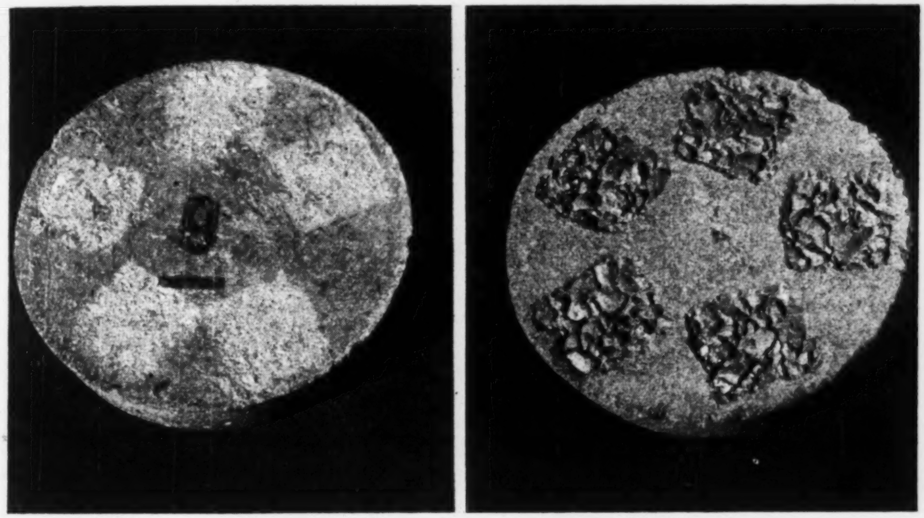
floors, because hardeners, paints, and the like are not used. However, investigations have shown that compressive strength is a very important factor. Data obtained indicate that abrasion resistance increases rapidly with a strength increase up to a certain point, depending upon aggregate, mix conditions, and type of test (**20, 23, 24**), but beyond this point increases in strength have very little effect on the abrasion resistance. This breaking point in the curve may come anywhere between 4000 and 6000 psi. Collins and Waters' data indicate that the initial rate of wear of 2000-psi concrete is about five times that of 4000-psi concrete. They further conclude that the type of aggregate used has an important effect on the later stages of wear of medium- and low-strength concrete. Above 6000 psi the type of aggregate has little effect on the wear resistance. Pogany (**25**) concludes that the abrasion resistance of concrete with hard dense aggregate bears no relation to abrasion resistance of the aggregate alone.

The ability of the road surface to resist abrasion depends primarily upon the mix, the placing, the finishing, and the curing of the concrete. Probably curing is the most important factor of those noted Improper curing can be very detrimental to the best designed and placed concrete as far as abrasion resistance is concerned. Tests by several investigators have shown the great differences in abrasion resistance caused by various types of curing and times of curing involved. It is a well established fact that the longer concrete is kept moist after the set has taken place, the more complete the hydration and the better the strength. Hence, it can be said that, in general, the abrasion resistance of ordinary concrete will be nearly proportional to its compressive strength, a fact that has been borne out by several investigations.

Since the introduction of the membrane type of curing compound, attention has been directed to the effect of such materials on the surface of the concrete. It has been found that some types of membrane material may react with the surface of the concrete in such a way as to inhibit or prevent proper hydration. This action, of course, weakens the surface and consequently reduces its wear resistance. Under these conditions, it is possible to use any of the three methods of test suggested to determine the resistance to abrasion. However, the dressing-wheel and shot-blast types are to be preferred over the rubbing type because the nature of the road surface is generally such that large aggregate is at or near the surface. Hence, by cutting the mortar, there will be a tendency to loosen the aggregate, thus simulating the action of chains or metal wheels on the road.

Another consideration in connection with membrane curing compounds and their effect on abrasion is the time of application. In this phase, the bleeding of the concrete is an important factor since it, too, has an effect on the surface and its resistance to abrasion.

There is a definite bleeding cycle dependent upon such things as water-cement ratio, aggregate grading, cement, temperature of the air, humidity, and wind velocity. Also, the use of purposefully entrained air will bring about changes in bleeding. These factors all have a direct bearing on the ability of the concrete to resist abrasion. Tests conducted on specimens that were treated with membrane curing compounds at various periods during the bleeding cycle have indicated that if they are applied before the concrete has stopped bleeding, the resultant surface has a relatively low abrasion resistance. In other words, the abrasion resistance of the concrete surface increases as the time of applying the curing compound to the concrete approaches the end of the bleeding period. There is little difference in the abrasion resistance if the curing material is applied between the end of the bleeding and the beginning of the drying of the concrete. However, once the concrete has started to lose water by drying after the bleeding has ceased, the abrasion resistance starts to decrease. This, of course, is due to lack of curing and is somewhat proportional to the degree of curing. The extreme is the difference in resistance of cured *versus* uncured concrete, as shown in Fig. 5. Burnett and Spindler (**22**) have also shown that the time of set, which generally coincides with the cessation of bleeding, is a critical point as far as the relationship of application of curing compound to abrasion resistance is concerned. Investigations of the abrasion resistance of concrete with regard to curing further illustrate the relationship of strength to abrasion. A poorly cured concrete will have low strength and low resistance to abrasion compared to concrete from the same batch that has been properly cured.



(*a*) Membrane curing compound. (*b*) No curing.

FIG. 5.—Two Specimens Made from the Same Concrete Mix Showing the Effects of Curing on Abrasion Resistance of Concrete.

Entrained air influences the resistance of the concrete to abrasion in about the same degree that it affects the strength of the concrete. Generally speaking, concretes containing not over 6 per cent entrained air will not show reduced resistance to abrasion as compared to non-air entrained concrete, provided the mix has been properly redesigned and all other conditions are equivalent (**23**). It has also been noted that concretes of the same compressive strength, although having a difference of as much as 10 per cent in air content, show approximately the same resistance to abrasion (**24**).

Other factors that tend to affect the abrasion resistance of concrete surfaces of roads are important but do not usually cause variations in the same project. Variations in the mix brought about by water-cement ratio, cement content, and aggregate grading all are reflected in the resistance to abrasion. Generally speaking, these variations follow the change in strength due to the above factors. The condition of the base on which the concrete is placed has an effect on the abrasion resistance insofar as the base influences bleeding. The finishing operation and the resulting fines brought to the surface by this operation will also have a marked effect on the resistance of the surface to abrasion by traffic.

Piles, Footings, Piers, etc.:

As previously stated, the abrasive forces that tend to erode concrete in water are developed by the movement of the water carrying foreign particles. These particles are generally sand and often may actually be softer than the concrete, but the force exerted by rapidly moving water carrying the sand is such that a cutting action is produced.

The most satisfactory method of duplicating this type of abrasion is obviously the shot blast. However, it is possible to evaluate the concrete to some degree by means of the dressing wheel because of the cutting action.

Concrete for this purpose is usable, even after the skin or surface has been worn off, except where appearance is an important factor. Therefore, in testing this type of concrete, consideration of its ability to resist abrasion is not only given to the surface but also to the basic concrete. After the surface has been cut away, the abrasive forces tend to cut the weaker portion of the concrete, which usually is the mortar, and destroy the bond of the aggregate, thus releasing it from the mass. The shot blast duplicates this action and therefore provides an accelerated method of test which cannot be duplicated satisfactorily by other methods. Kennedy (23) and Witte and Backstrom (24) have reported results by this method.

OTHER TEST METHODS

Many other methods of test than the three mentioned up to this point have been used by investigators with varying results.

One such test involved a modification of the Los Angeles rattler (7, 8). Concrete cylinders or cubes were placed in the machine and tumbled for various periods of time. The abrasion resistance was then determined by visual observation and determination of weight loss. This is a rather severe test, involving a pounding action not commonly associated with abrasion. A hard, brittle concrete might break up in this test, and a softer material might stand up. In actual field practice, however, the harder concrete would resist abrasive forces much better than the relatively softer material. Hence, it is not believed that this method is well suited for the determination of abrasion resistance under such conditions as set forth in this paper.

Another method of determining abrasion resistance is by means of an abrasive wheel. A carborundum or similar wheel, while moving, is brought into contact with the concrete surface. A constant pressure is applied and a constant time of application is used. The specimen may be either wet or dry and generally provision is made to remove the abraded material during the test interval. This method is weak because of variations in grinding wheels and variations in individual wheels as they become worn. The wheel also will be supported by the toughest portion of concrete with which it comes in contact and, therefore, measures the abrasion resistance of the strongest rather than the weakest link.

Several methods involving balls, shoes, and rolls have been used experimentally with varying degrees of success. One of these, a ball method proposed by R. E. Davis of the University of California, develops wear by rolling steel grinding media under pressure over the surface of the concrete. Provision is made to avoid tracking of the balls. The surface of the concrete is subjected to flowing water to wash the abraded material off as it is produced. This apparatus is rather bulky and costly, factors which are a disadvantage to the method. Also, tests to date have not indicated that the results obtained are appreciably more consistent or representative than those with other less expensive pieces of equipment.

There are probably many other methods which have not come to the authors' attention, but it is believed that those that have been mentioned are typical of the various types.

The losses due to abrasion are generally expressed as percentage weight loss or depth of wear. Either one or both are applicable to all test methods and may be determined by various means.

CONCLUSIONS

The results of the investigations to date have indicated that there is no one method of test for abrasion resistance that will satisfy all conditions. The factors involved in the various types of wear on various concrete surfaces preclude the use of a single type of test. However, it appears possible that a combination of two types, such as the shot blast and rubbing or shot blast and dressing wheel, may be worked out as a practical solution of the problem.

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DISCUSSION

CHAIRMAN A. T. GOLDBECK.[1]—Some years ago, in fact too many years ago, I had something to do with an abrasion test—a sand blast test on paving brick, using crushed quartz sand. I was interested in Mr. Kennedy's remark on the effect of coating the specimen with a rubber coating.

We used a rubber pad with a hole in it to control the position in which the abrasion took place. That rubber pad outlasted many, many specimens of brick. When the specimen is coated with rubber, the deceleration of the particles striking the surface is reduced and thereby the striking force is reduced.

We have had other occasions to use abrasion tests. The old Dorry Hardness Test was nothing more than an abrasion test. It was designed about 75 yr ago in the French School of Bridges and Roads for testing the hardness of rock. Some years ago we used a wear test involving a number of emery wheel cutters mounted on a horizontal axis and subjected to a given load for a given number of revolutions.

That device certainly wore the surface of the specimen. I do not think much of it as a test, however, because the emery wheel cutter is not designed for that purpose. It has a variable contact area as the cutter wears down, but if it could be devised so that the teeth were of constant cross-section, I think it would make a pretty good test.

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[1] Engineering Director, National Crushed Stone Assn., Washington, D. C.

PULSE VELOCITY TESTING OF CONCRETE*

By W. E. Parker[1]

For some seven years members of the research staff of the Hydro-Electric Power Commission of Ontario have been engaged in the development and application of apparatus for the nondestructive testing of concrete. The pulse velocity equipment and techniques that were adopted because of their suitability to the needs of the commission have been described on previous occasions before this and other societies (**1, 2, 3, 4**).[2] In addition, the experiences of others in using pulse propagation techniques for testing concrete have been recorded (**5–15**). This paper is intended mainly as a review of the continuing use of the Soniscope by the commission, showing the fields in which it is finding its greatest usefulness and indicating the limitations that have been encountered. The applications that will be described include surveys of cracks in massive hydraulic structures, durability or condition surveys of old structures, the study of early strength development in concrete, and laboratory studies involving its elastic and mechanical properties.

APPARATUS

Since the Soniscope equipment has been described in detail in earlier papers (**1, 2**), it is necessary here to mention only the main components and the general method of operation. The unit consists chiefly of an electronic generator of repetitive electrical signals, a multiple-crystal transmitter to introduce ultrasonic pulses into the concrete, and a similar crystal receiver. Both the transmitted and the received pulses are displayed on a time base on the screen of a cathode-ray tube from which the time of pulse travel can be determined readily. From the time interval shown by the Soniscope and the distance between transducers, the pulse velocity between them is calculated.

In use the transducers are placed preferably against opposite faces of a unit, although signals may be transmitted for moderate distances when both transducers are applied to points on the same surface. It has become the general practice of the Hydro-Electric Power Commission of Ontario, as it has with many other Soniscope users, to deal in terms of comparative velocity values directly rather than to attempt to calculate the dynamic modulus of elasticity. This avoids the introduction of density and Poisson's ratio, both of which are difficult to determine for concrete *in situ*. Thus, in this regard, our views are in agreement with those expressed by Whitehurst (**6**).

SURVEYS OF CRACKS

As was pointed out in earlier papers (**1, 2**), the pulse velocity technique as developed by the commission was directed primarily towards the evaluation of surface cracking in major gravity dams. Interest in surface cracking was keen in the immediate post-war years when the high-lift method of dam construction which had proved satisfactory for smaller structures was being used for progressively taller dams. As a consequence the behavior patterns of cracks which sometimes appear in most major structures shortly after form removal have been studied in some detail. After demonstrating agreement with the depths indicated by an earlier dye-injection method (**16**), the pulse velocity method was adopted because of its superior speed and convenience. Eight dams ranging in height from 100 to 235 ft have been surveyed in whole or in part using the pulse technique. Repeated observations have shown that surface cracks which appear shortly after the concrete is exposed may have initial average depths as great as 1½ ft, but these usually close significantly within a few months and continue to become shallower, although at a decreasing rate in succeeding years (Fig. 1).

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Research Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Can.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1041.

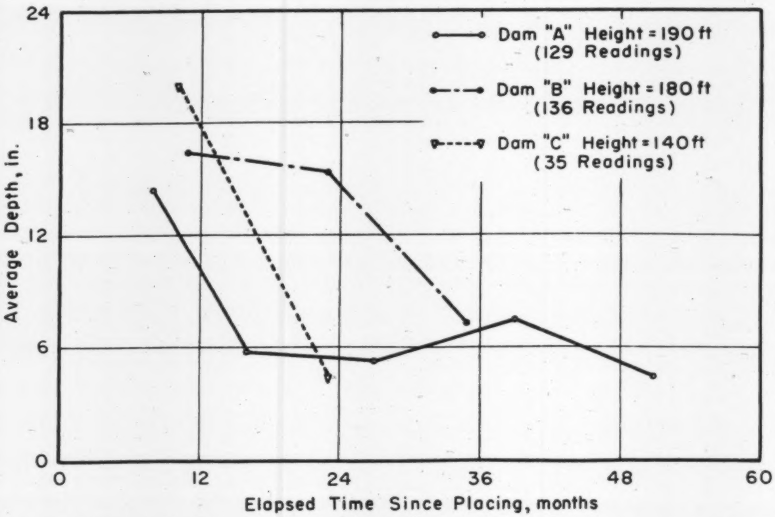


FIG. 1.—Depth of Surface Cracks in Gravity Dams as Revealed by Pulse Velocity Surveys.

Some cracks intersecting the access tunnels near their portals have been difficult to trace because of long transmission paths and a heavy curtain of reinforcing, but these have been shown to be restricted at least to the lifts in which they occurred.

Since questions have arisen regarding the possibility of cracks existing undetected in the interior of gravity sections, this possibility has been investigated by installing remote-reading embedded instruments and by pulse transmission through the structure. In the larger dams the search for such possible internal cracks is frequently hindered by the limiting path distance of 50 to 60 ft. One method that has been adopted to overcome this limitation is the drilling of exploratory holes at a spacing of 40 ft and the use of special transducers that will function in the 6-in. diameter holes. Successful transmission between such holes drilled recently in a 200-ft high dam lends support to the negative indications given by crack meters and other instruments embedded in the same structure.

CONDITION SURVEYS

Many applications have been found for pulse velocity measurements as a means of indicating the condition of structures. Condition may be considered from the standpoint of deterioration or of inhomogeneities resulting from segregation or lack of compaction. Examples of studies of deterioration caused by weathering, leaching, freezing during early hardening, and exposure to fire are described in the following paragraphs.

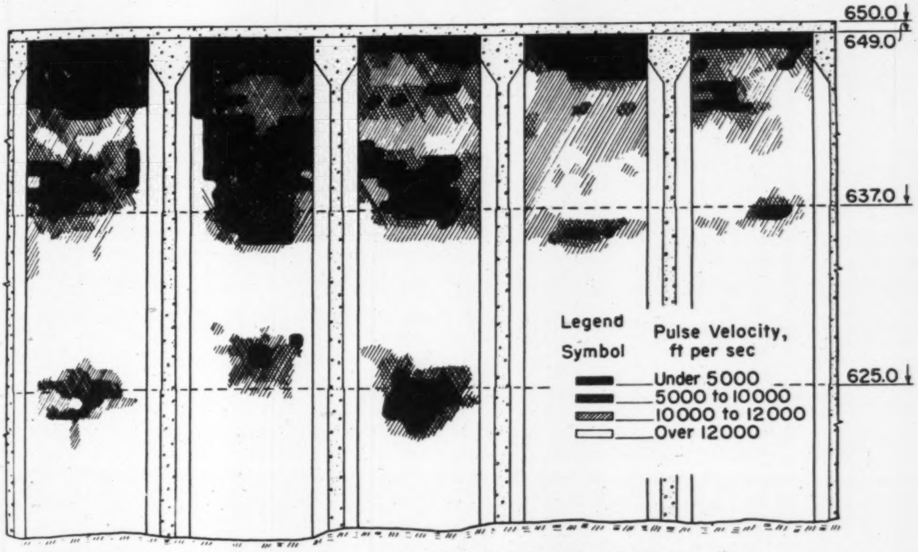


FIG. 2.—Outline of Deterioration in Slabs of a Reinforced Concrete Dam.

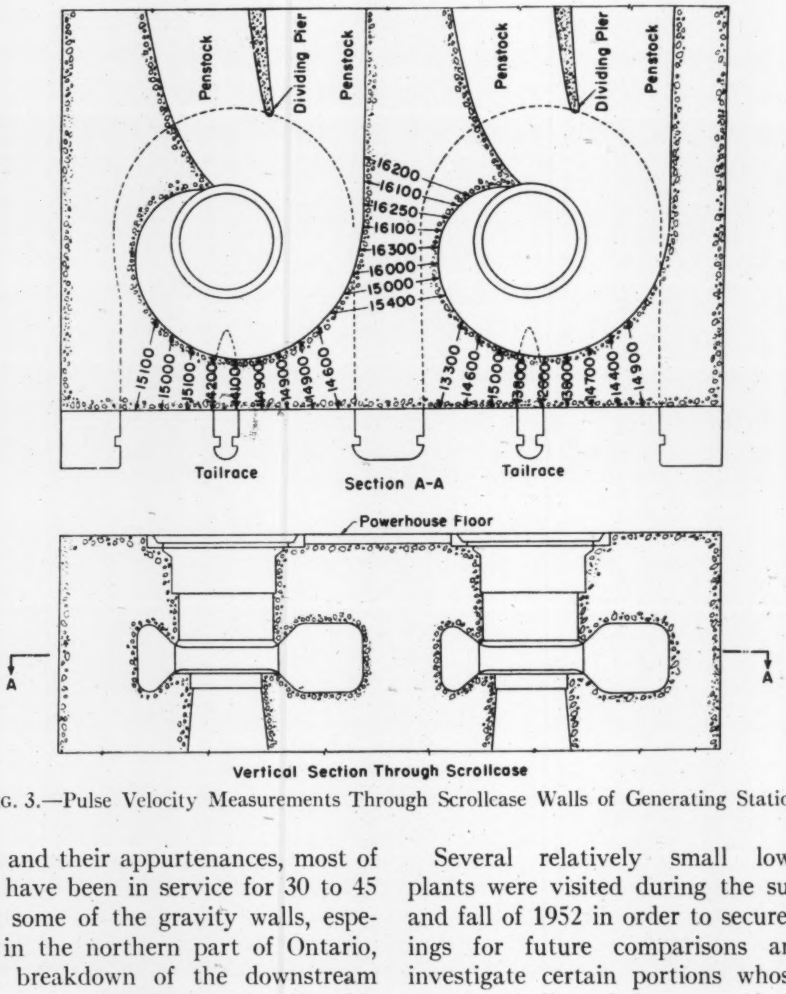
Slab-and-Buttress Dam:

One of the earliest deterioration surveys carried out by the commission dealt with a dam built in 1914. Some 70 slabs and an equal number of buttresses and haunches were investigated by means of the Soniscope; these required a total of nearly 50,000 readings, most of them taken at a spacing of 1 ft. A three-man party, assisted by two laborers to handle ladders and scaffolding, completed this survey in about 2 months. Much of the survey time was occupied in locating the observation points and in shifting the ladders. The apparent pulse velocities as measured in the structure ranged from below 5000 to over 17,000 ft per sec. A typical picture of velocity distribution for one of the slabs is shown in Fig. 2. This indicates how deterioration has progressed from some of the horizontal construction joints. The urgency of repairs in each slab is, of course, related to the size and location of the zones of severe deterioration and also to the extent of the more moderately affected regions.

Assistance in assessing the significance of various velocity levels was provided by establishing velocity values for the best preserved portions of the structure and by removing 2-in. diameter cores at key points. Although there was too much breakage in the cores taken from low-velocity zones to provide a clear relationship between velocity and strength, there was good agreement between velocity and the characteristics of concrete as revealed by core breakage and rate of drilling.

Other Hydraulic Structures:

On a number of occasions Soniscope readings have been made on gravity-type walls and on portions of hydraulic power plants and their appurtenances, most of which have been in service for 30 to 45 yr. In some of the gravity walls, especially in the northern part of Ontario, severe breakdown of the downstream face may be found. Occasionally this concrete is disintegrated to such an extent that pulse transmission through it is impossible. However, it is usually found that the disintegrated layer is only a few inches thick and that the remainder of the wall is sound as indicated by satisfactorily high pulse velocities. This has been demonstrated by chipping the weakened layer away or by driving in chisels or railroad spikes until good transducer contact is established with the sounder underlying concrete.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14400
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Tailrace
Section A-A
Tailrace
Powerhouse Floor
A
A
Vertical Section Through Scrollcase

FIG. 3.—Pulse Velocity Measurements Through Scrollcase Walls of Generating Station.

Several relatively small low-head plants were visited during the summer and fall of 1952 in order to secure readings for future comparisons and to investigate certain portions whose appearance indicated that some deterioration might have occurred. Since the plants were in operation, only limited parts of these structures could be examined successfully; brief shutdowns for routine inspection and maintenance provided the only opportunities for examination of underwater parts. Some indication of the difficulties involved in "shooting" through the curved, sloping walls of penstocks and scrollcases is given by Fig. 3. In particular, there is need for accurate location of points of transducer application so that the apparent velocity is not unduly affected by errors in the calculation of path distance. In general, the condition of the concrete in the lower parts of the plants was found superior to that above water level. Trouble was noted in the downstream wall of some plants at the level of the scrollcase and could be traced to the lack of attention given to horizontal joints in the days before thorough concrete control was exercised.

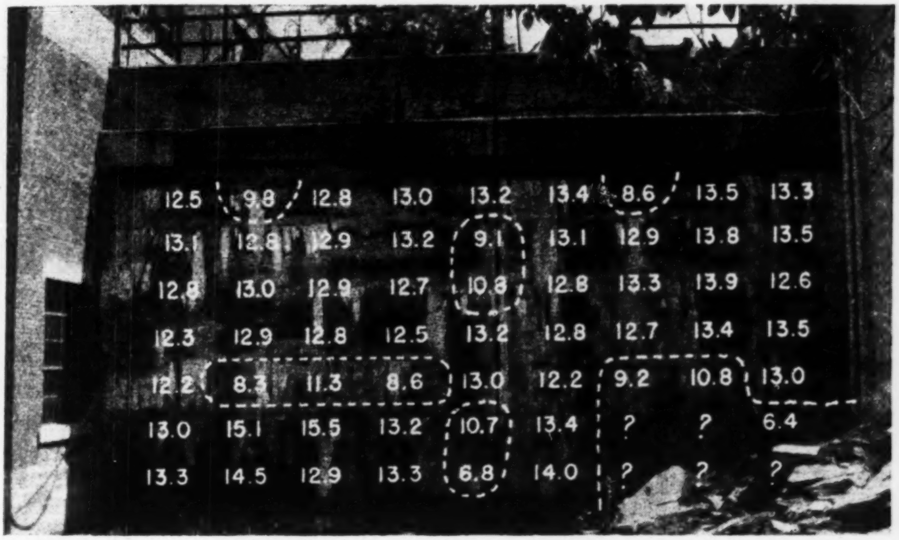

FIG. 4.—Disclosure of Weakening Portions of Intake Wall by Soniscope Survey.

Included in one of the power plant surveys was the intake wall shown in Fig. 4. The velocity "contours" that are shown superimposed on the photograph indicate a pattern of deterioration that could not have been deduced from the appearance of the surface.

Switchyard Concrete:

An interesting study of the distressed condition of certain piers in a switching station was carried out recently. Pulse velocity measurements taken as shown in Fig. 5 showed that the trouble originated at the top and side surfaces. Repeated measurements, after another winter's exposure, revealed significantly advanced deterioration in some instances. This example tends to show the usefulness of pulse velocity surveys for disclosing, from a single survey, uniformity throughout a structure and, from repeated observations, the progress of deterioration.

Concrete Buildings:

Accidental damage to concrete during building construction may sometimes be mapped out rapidly by a pulse velocity survey. In one instance the extent of fire damage to concrete columns shortly after form removal was readily ascertained. Interpretation of the velocity readings was achieved by determining the velocity-strength relationship of a group of control cylinders tested at various ages. Another successful study of fire damage was reported by Andersen and Nerenst (9).

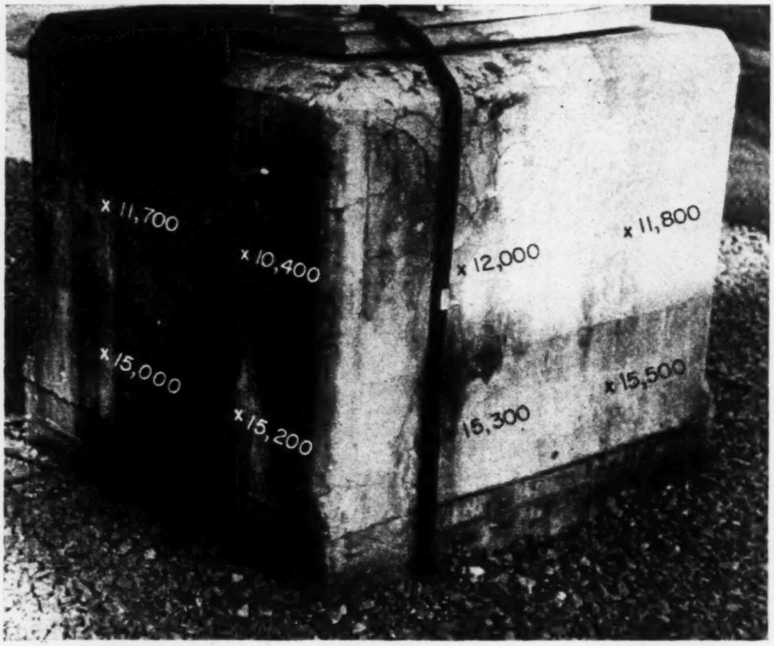

FIG. 5.—Extent of Deterioration in Small Pier as Shown by Pulse Velocity Readings.

Numerous instances have occurred in which laminated conditions could be detected by low velocities. The extent to which a floor topping had loosened from the base slab was disclosed readily in a situation where the underside of the slab was accessible and so permitted transmission through from top to bottom. Similarly, the gradual separation of the elements of a sandwich slab having a cellular glass core was successfully observed as the slab samples underwent severe freezing-and-thawing exposure. Laboratory measurements on a section of a Gunite structure that had failed by lamination showed significantly lower velocities in the direction at right angles to the surface than parallel to it. The limits of frost damage producing a similar type of failure in a floor slab were also disclosed by a Soniscope survey. The damage had evidently occurred as the result of inadequate protection during a prolonged period of cold weather that occurred shortly after placing.

Pavements:

Because of the commission's limited interest in concrete pavements, the only such survey carried out was the one reported by Leslie and Cheesman (1). This indicated that velocities might range from over 16,000 to about 6000 ft per sec, and these seemed to agree reasonably well with the appearance of the roads. Since that time Whitehurst (7), Andersen and Nerenst (**9**), and Meyer (**15**) have reported more extensive work of this nature. None of these authors has reported velocities as low as might be expected from the poor surface condition of some of the roads tested. From these experiences it appears that difficulties are likely to be encountered more frequently in the interpretation of pulse velocity data on pavements than on most other concrete structures.

CONCRETE SETTING AND HARDENING

Setting Behavior:

Studies of the changes in properties associated with the setting of concrete have been described by Whitehurst (**7**) and Cheesman (**8**) and discussed by Andersen and Nerenst (**9**), Jones (**13**), and L'Hermite (**14**). Curves of pulse velocity *versus* time have usually shown a rapid rise during the stiffening period followed by a decreasingly rapid rise about the time when final set would have occurred in the neat cement.

In measurements on relatively stiff concretes, which would permit the removal of forms shortly after molding, it has been possible, as shown by Whitehurst, to apply transducers directly to the concrete and to obtain well-defined points of inflection. But in dealing with plastic concrete or with high-slump concrete, the mechanics of measurement become more difficult because the forms must be left in place. Embedment of transducers has been successful, but this procedure limits the number of conditions that can be studied simultaneously. Rubber diaphragms and removable ports in the forms have allowed observations to be made, although it has been felt that some inaccuracies result. High rates of attenuation of the pulse in fresh concrete either place serious restrictions on the path length or result in poorly-defined received signals owing to distortion of the original wave form. Certain measurements made in the commission laboratory on plastic concretes have failed to disclose a clear point of inflection corresponding to final set. It is believed that Bureau of Reclamation tests also show no clear point of inflection.

It appears, therefore, that the Soniscope method offers a means of studying the setting characteristics of concrete but that further refinements in technique are desirable in dealing with the wetter mixes.

Strength Development:

There has naturally been a good deal of speculation regarding the possibility of predicting the test strength of concrete from pulse velocity measurements. The earliest commission data were reported by Cheesman (**8**). From these it was believed to be feasible to estimate the compressive strength with a useful degree of accuracy only in the very low ranges. However, this suggested a field of application in checking the safety of shore or form removal and of detecting lack of uniformity of the concrete in a structure.

In a later series of proportioning tests for a large construction project, the pulse velocity of all specimens was determined immediately before the compression test. On this occasion, however, the velocity was measured with somewhat greater precision by using the 12-in. axial path rather than a 6-in. diameter as was done in the earlier series. Only one type of aggregate was used. Two sources of type I cement were included as well as three kinds of blended cement. Admixtures investigated were air-entraining, accelerating, and water-reducing types. In studying the curves plotted for each of the 46 mixes, no significant differences in the pulse velocity *versus* strength relationships were detected and hence the data reflected, quite effectively, the different rates of early strength development.

A correlation analysis of all the data treated without regard to differences in cements, admixtures, and curing conditions yielded a linear relationship between pulse velocity and the logarithm of compressive strength. From this the test strength of specimens could be predicted from velocity readings with the following degrees of certainty:

Pulse Velocity, ft per sec	Estimated Strength (Mode), psi	Lower 95 per cent Confidence Limits, psi[a]
9 000	184	85
10 000	292	135
11 000	460	213
12 000	730	337
13 000	1150	533
14 000	1800	842
15 000	2840	1329
16 000	4440	2099

[a] These upper and lower confidence limits define a zone within which results may be expected to fall 95 per cent of the time.

Owing to skewness, the upper confidence limits were between four and five times as great as the lower limits. However, in applications where it is necessary to insure only a minimum strength requirement, the magnitude of the upper confidence limits does not present a serious limitation. For some purposes, then, the prediction of compressive strength from pulse velocity may have practical value at levels below 1000 or 1500 psi where the possible errors are not likely to be greater than a few hundred pounds per square inch. However, in the usual range of working strengths (from 2000 to 5000 psi), possible errors in prediction which increase correspondingly from 1000 to 2000 psi or more are usually far too great to clearly disclose anything less than gross strength variations.

Although the above analysis shows a somewhat disappointing picture, the accuracy of a strength estimate for a portion of a given structure will probably be somewhat greater. This may be ascribed to the elimination of the many mix variations that were included in the above estimate and to the longer path distance that is usually possible. In addition, the use of pulse velocity on concrete *in situ* does not involve the variance introduced by individual compression test values. Accordingly, as Jones (13) has demonstrated, pulse velocity measurements are finding a number of useful applications in establishing acceptable strength limits.

LABORATORY TESTING

Where pulse velocity apparatus is available in a laboratory, it will soon be found to have many uses, especially in supplementing the information obtainable from other tests.

In detecting and charting the course of breakdown of durability specimens, the test shows some advantages over the resonant frequency method, especially from the standpoint of speed and ability to check the condition of a specimen along several paths. Although the internal changes in specimens exposed to freezing-and-thawing conditions are detected readily by reductions in pulse velocity, limited data suggest that the expansion produced by certain aggregates in the wetting-drying, heating-cooling test does not bring about correspondingly great changes in velocity.

While all organizations engaged in pulse velocity testing have investigated to some extent the effects on pulse velocity of factors such as aggregate and cement type, mix proportions, moisture content, admixtures, and reinforcing, the results reported at various times have not all been in full agreement. However, the extensive work in this field that was reported recently by Jones (13) should serve as a valuable reference for some time to come. The tests, which were carried out with relatively high-frequency, short-range apparatus, seem to reflect a higher degree of accuracy of velocity measurement than similar work with the standard Soniscope. This may help to explain an occasional lack of conformity between Jones' findings and Soniscope findings in certain laboratory studies.

CONCLUSIONS

1. The limit of transmission distance ranges from a few inches in incoherent concrete, which is incompletely set or is seriously deteriorated, to a maximum of 50 or 60 ft in concrete of high quality.

2. The depth of surface cracks in major structures may be ascertained with suitable observational techniques.

3. The absence of internal cracking in mass concrete is indicated by pulse transmission through the mass at normal velocity; the presence of such cracks would be suggested by absence of a received signal or abnormal delay in its arrival.

4. Deterioration may be clearly detected in either structures or in laboratory freezing-and-thawing specimens. Where possible, interpretation of the data may be enhanced by earlier reference measurements and by supplementary information such as may be provided by coring, physical examination, and testing.

5. Within limitations imposed by high attenuation and form interference, the setting behavior of concrete may be studied.

6. Compressive strength can only be approximated even with known materials. However, the accuracy obtainable may be adequate for controlling the removal of forms and for revealing serious inadequacies of concrete *in situ*.

From the foregoing it has been shown that pulse velocity techniques offer many possibilities for the rapid, non-destructive testing of concrete either in the laboratory or in field structures. The known limitations in application and interpretation have been pointed out and even emphasized in order to discourage undue optimism by potential users. However, the commission's experience over a period of years with several Soniscope units has established a high degree of confidence in the indications provided by pulse velocity measurements, especially when these are obtained by crews trained in observation and interpretation. It is to be expected that the future will bring to the method new uses and greater discriminating power in its present applications.

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(13) R. Jones, "The Testing of Concrete by an Ultrasonic Pulse Technique," *Proceedings*, Highway Research Board Vol. 32, p. 258 (1953).

(14) J. Chefdeville and G. Dawance, "Dynamic Sounding of Concrete," *Annales*, L'Institut Technique du Batiment et des Travaux Publics, No. 140, July–August, 1950 (introduction by R. L'Hermite).

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DISCUSSION

Mr. R. Jones[1] (*by letter*).—Mr. Parker has presented an excellent brief summary of the use of the pulse technique for testing concrete. The work of the Hydro Electric Power Commission on dams and other large structures provides admirable examples of the practical application of the technique.

At the Road Research Laboratory, England, we have been more concerned with smaller distances of propagation, and our work suggests that the technique is also applicable to testing new structures. In such favorable conditions, the strength *in situ* can be obtained to a much better accuracy than is possible from conventional static tests.[2]

Our major problem is to apply the technique to road pavements, but here, as with Mr. Parker, we find difficulty in interpreting the results. Measurements of the wave velocity along the surface of the pavement do not reveal any changes in density which may occur with depth in poorly compacted concrete. Even in well-compacted concrete we have found a discrepancy between the wave velocity along the surface and through cores cut from the pavement. We have always aimed at using a reflection technique on pavements, and with the advent of barium titanate transducers this becomes a distinct possibility.[3] The National Physical Laboratory has reported successful pulse reflection in small samples 6 to 18 in. thick under ideal conditions when top and bottom surfaces were smooth and parallel. Our subsequent experience on pavements *in situ* suggests that any unevenness on the underside will be a severe handicap. In consequence, we are investigating the possibility of using sustained vibrations of low frequency as suggested by Gerald Pickett.[4]

Mr. W. E. Parker (*author's closure*).—I agree that for short path distances the higher frequency propagation adopted by the Road Research Laboratory may achieve greater discrimination than is possible with the longer-range Soniscope apparatus. Hence, Mr. Jones will undoubtedly experience greater success in predicting strength of concrete *in situ* within the operating range of this form of the apparatus than was indicated for the Soniscope.

The pulse reflection method, if present difficulties can be overcome, would be of great value in many instances. It is encouraging to learn that some measure of success has been attained with this method, even if only under ideal conditions. Although it seems probable that the sustained vibration technique will be limited somewhat by boundary effects it is interesting to learn that the possibilities of the method are now being actively investigated.

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[1] Road Research Laboratory, Department of Scientific and Industrial Research, Harmondsworth, England.

[2] R. Jones, "The Testing of Concrete by an Ultrasonic Pulse Technique," *Proceedings*, Highway Research Board, Vol. 32, pp. 258–275 (1953).

[3] G. Bradfield, unpublished data.

[4] G. Pickett, "The Dynamic Testing of Pavements," *Journal*, Am. Concrete Inst., Vol. 16, No. 5, pp. 473–489 (1945).

DETERMINATION OF COMPRESSIVE STRENGTH OF CONCRETE BY USING ITS SONIC PROPERTIES*

By Clyde E. Kesler[1] and Yoshiro Higuchi[2]

Synopsis

In predicting the quality of concrete by sonic methods, the prediction is usually based on the initial modulus of elasticity; however, considering concrete as viscoelastic, an analysis of a rheological model of concrete clearly shows that a property in addition to the initial modulus of elasticity must be considered. The one property, other than modulus of elasticity, that promises to be of greatest help in predicting the strength of concrete is the coefficient of viscosity. While this property may be an excellent one to use, it cannot be determined readily from sonic tests of concrete; but, logarithmic decrement, which is related to the coefficient of viscosity, can be easily obtained from sonic tests.

To establish whether or not there exists a relationship between the modulus of elasticity and damping capacity of concrete and the compressive strength of concrete, some 300 standard 6 by 12 in. cylinders were tested. From the test data a set of curves is obtained from which the strength of concrete, made of aggregates used in this investigation, can be predicted. The accuracy of the prediction is generally within an error of 5 per cent. This accuracy can be obtained without knowledge of the age, mix, or moisture content of the concrete. The results clearly show why the use of the modulus of elasticity alone is not sufficient to predict accurately the strength of concrete.

In the study of plain concrete, it is often desirable to be able to determine certain physical properties without subjecting the specimen to a destructive test or even to stresses higher than a small percentage of the ultimate stress. The determination of properties such as the dynamic modulus of elasticity and the damping capacity may be of particular importance in the study of fatigue, creep, and durability of concrete. It is desirable in the type of studies just mentioned to determine the physical properties of the same specimens upon which the studies are being conducted; and of course, in order to do this, a non-destructive means of testing must be used. Sonic testing of concrete has been recognized for several years as a useful and powerful non-destructive means for studying the quality of concrete. The object of the investigation discussed in this paper was to determine what sonic properties of concrete, if any, would enable one to predict accurately the compressive strength of the concrete.

Engineers have believed for many

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years that a relationship exists between the modulus of elasticity and compressive strength of concrete. Several experimental formulas have been presented giving such a relationship; however, it is possible to be more than a hundred per cent in error if these formulas are used for material or conditions for which they were not derived. Thus, it becomes clear that if the compressive strength of concrete is to be predicted with any degree of accuracy, some physical property in addition to the modulus of elasticity must be used. Furthermore this additional property must be one which can be obtained in a non-destructive test.

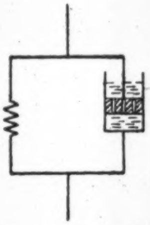

FIG. 1.—Simplest Mechanical Model Which Can Be Considered to Represent Concrete.

If an analysis is made of the rheological model shown in Fig. 1, which is the simplest model which may be assumed to represent concrete, it becomes clear that damping properties should be taken into consideration as well as the elastic properties. Thus, it would seem clear that knowledge of the coefficient of viscosity would be extremely useful in predicting the behavior of concrete. Although this property cannot be determined readily from sonic tests of concrete, the logarithmic decrement, which is a measure of the specific damping capacity and is related to the coefficient of viscosity, can easily be obtained from sonic tests.

The dynamic modulus of elasticity may be determined from the resonant frequency of the specimen and may be computed by the following equation:[3]

$$E_D = Cwf_0^{\,2} \ldots\ldots\ldots\ldots (1)$$

where:

E_D = dynamic modulus of elasticity,
f_0 = resonant frequency,
w = weight of specimen, and
C = a factor which depends upon the shape and size of specimen, the mode of vibration, and Poisson's ratio.

Graphs are given by Pickett[3] for determining the size and shape factor C.

While the logarithmic decrement may be obtained in several ways, it may be obtained most easily by measuring the sharpness of the resonance curve and may be computed from the following approximate equation:[4]

$$\delta = \pi \frac{(f_2 - f_1)}{f_0} \ldots\ldots\ldots\ldots (2)$$

where:

δ = logarithmic decrement,
f_0 = resonant frequency, and
f_1, f_2 = frequencies on either side of resonance at which the amplitude of vibration is 0.707 of the maximum.

This method of obtaining the logarithmic decrement was chosen because of its simplicity.

APPARATUS

A schematic diagram of the apparatus used in this investigation is shown in Fig. 2. All equipment was selected and used so as to meet the requirements of the ASTM Tentative Method of Test for Fundamental Transverse and Torsional Frequency of Concrete Specimens

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[3] Gerald Pickett, "Equations for Computing Elastic Constants from Flexural and Torsional Resonant Frequencies of Vibration of Prisms and Cylinders," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 846 (1945).

[4] W. T. Thomson, "Measuring Changes in Physical Properties of Concrete by the Dynamic Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1113 (1940).

(C 215 T).[5] The oscillator is a Hewlett-Packard Model 200-I tuned circuit type. It covers a frequency range of 6 to 6000 cps and delivers approximately 10 v. The oscillator feeds a Knight 10-w power amplifier which in turn controls the driver.

The driver consists of a standard Jensen 8-in. speaker modified as follows: the protecting felt covering over the voice coil was removed and a small aluminum rod was cemented to the voice coil. A length of $\frac{1}{4}$-in. round aluminum rod was then attached to the first rod to act as a driving rod. The driving rod was initially supported at the face of the speaker by a thin leather diaphragm. However, in the belief that excessive damping in the speaker may have been produced by this leather support, it was removed after the tests had begun and was replaced by two very thin brass straps. There was no noticeable change of damping in the speaker. As an aid to eliminating damping and also to reduce the noise, a major part of the cone was removed. However, sufficient cone was left to support adequately the voice coil.

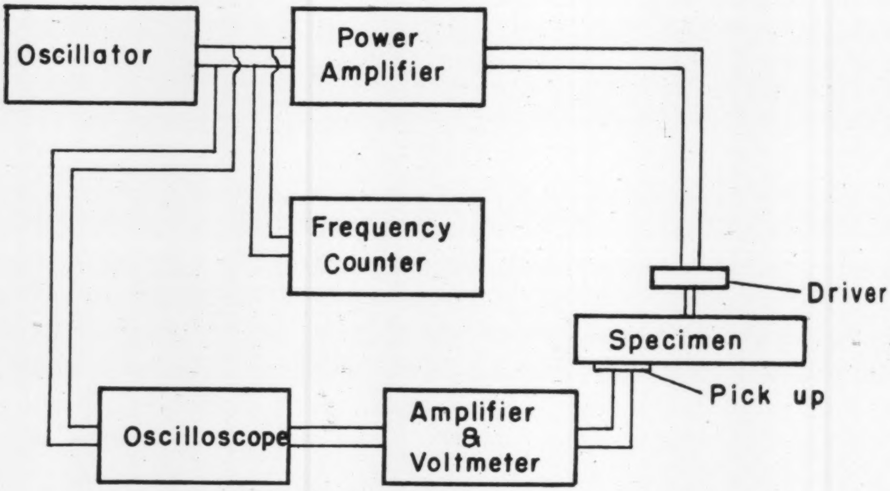

FIG. 2.—Schematic Diagram of Apparatus.

The specimen is vibrated by placing the $\frac{1}{4}$-in. aluminum rod in firm contact with it. The vibration of the specimen excites a small crystal pickup in a Brush model KN-1 "Vibromike," which is a contact microphone of the inertia type with a frequency range from 30 to 6000 cps. Below 1000 cps the pickup delivers a voltage proportional to the acceleration it receives from the vibrating specimen. Above 1000 cps the voltage delivered is proportional to the velocity of the vibrating body.

Amplification for the pickup and a meter for indicating the strength of the signal picked up are combined in a single unit, Hewlett-Packard Model 400-C, vacuum tube voltmeter. It has a frequency range from 20 to 2,000,000 cps. The amplifier provides a gain of approximately 50 decibels. A DuMont 274-A cathode ray oscilloscope with A5BP1A cathode ray tube is used with this equipment more as a matter of convenience than necessity. The oscilloscope may be

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[5] 1952 Book of ASTM Standards, Part 3, p. 1072.

TABLE I.—MIXES, CURING, AND TEST RESULTS.

Series	Mix by Weight Cement Sand Gravel	Water-Cement Ratio by Weight	Cured		Tested	Age, days	E_D, psi	δ	f'_c, psi
			Moist, days	Dry, days					
IA	1:1.72:2.85	0.467	7		Wet	7	5.16 × 10^6	0.049	3920
			7	7	Dry	14	5.17	0.033	4760
			7	21	Dry	28	4.84	0.029	4720
			7	84	Dry	91	4.63	0.023	5000
IB	1:1.74:2.87	0.446	7		Wet	7	5.31	0.050	4390
			14		Wet	14	5.52	0.044	4590
			28		Wet	28	5.92	0.041	5430
			91		Wet	91	6.25	0.038	6080
			7	7	Dry	14	5.30	0.031	4890
			7	21	Dry	28	5.32	0.026	5930
			7	84	Dry	91	5.20	0.023	5870
			7	1	Dry	8	5.20	0.040	4340
IC	1:1.77:2.92	0.420	7		Wet	7	5.54	0.046	5270
			7	7	Dry	14	5.48	0.031	5520
			7	21	Dry	28	5.35	0.027	6970
			7	84	Dry	91	5.05	0.023	6790
IIA	1:2.28:3.31	0.555	7		Wet	7	4.89	0.053	3410
			7	7	Dry	14	5.18	0.033	4530
			7	21	Dry	28	4.98	0.028	5070
			7	84	Dry	91	4.94	0.023	5370
			7	1	Dry	8	5.05	0.042	3640
			7	1	Wet	9[a]	5.07	0.054	3690
IIB	1:2.31:3.34	0.530	7		Wet	7	5.05	0.052	3610
			14		Wet	14	5.40	0.047	4520
			28		Wet	28	5.71	0.044	4830
			91		Wet	91	6.01	0.040	5390
			7	7	Dry	14	5.21	0.035	4380
			7	21	Dry	28	5.38	0.027	5530
			7	84	Dry	91	5.08	0.022	5720
IIC	1:2.33:3.39	0.498	7		Wet	7	5.50	0.050	4240
			7	7	Dry	14	5.42	0.032	5200
			7	21	Dry	28	5.27	0.028	5460
			7	84	Dry	91	5.08	0.024	5710
			7	1	Dry	8	5.16	0.039	3700
			7	1	Wet	9[a]	5.27	0.047	3700
IIIA	1:3.14:3.93	0.681	7		Wet	7	4.69	0.055	2980
			7	7	Dry	14	4.71	0.038	3550
			7	21	Dry	28	4.81	0.030	4110
			7	84	Dry	91	4.64	0.025	4040
IIIB	1:3.17:3.96	0.651	7		Wet	7	4.83	0.054	2860
			14		Wet	14	5.22	0.048	3920
			28		Wet	28	5.28	0.046	4040
			91		Wet	91	5.59	0.043	4370
			7	7	Dry	14	4.83	0.034	3580
			7	21	Dry	28	4.77	0.028	4120
			7	84	Dry	91	4.55	0.023	4240
			7	1	Dry	8	4.82	0.049	2730
IIIC	1:3.22:4.03	0.612	7		Wet	7	5.03	0.053	3350
			7	7	Dry	14	5.10	0.034	4160
			7	21	Dry	28	5.03	0.029	4260
			7	84	Dry	91	4.94	0.023	4980
IVA	1:3.79:5.28	0.860	7		Wet	7	4.02	0.059	1390
			7	7	Dry	14	4.14	0.042	1710
			7	21	Dry	28	4.13	0.034	2070
			7	84	Dry	91	3.97	0.021	2680
VC	1:4.36:6.19	0.860	7		Wet	7	4.65	0.053	1890
			14		Wet	14	4.92	0.050	2300
			28		Wet	28	5.15	0.052	2560
			91		Wet	91	5.43	0.046	2940
			7	21	Dry	28	4.18	0.035	2200
			7	84	Dry	91	4.24	0.025	2470

[a] Soaked in water one day after being cured seven days moist and one day dry.

used as an aid in checking the other equipment and is most frequently used to indicate the mode of vibration of the specimen.

Because of the accuracy required in determining the quantity $(f_2 - f_1)$ of Eq 2, the frequencies are determined by use of a Berkley Eput Meter Model 554B. With the use of this frequency counter it is possible to measure the frequencies from the oscillator to approximately $\frac{1}{10}$ cps. However, this accuracy was not needed from a practical standpoint, and the frequencies were determined only to the nearest cycle. Equipment by other manufacturers may be used with equal facility. The particular components of this apparatus were used because of their availability.

The relative error in determining the modulus of elasticity, E_D, caused by using incorrect frequency readings is, from Eq 1:

$$\frac{dE_D}{E_D} = \frac{dC}{C} + \frac{dw}{w} + \frac{2df_0}{f_0}.$$

This equation shows that for an error of 0.5 per cent in reading f_0, the error in E_D will be 1 per cent. The scales of most oscillators are marked in such a manner that it is not difficult to read them within 0.5 per cent error and a 1 per cent error is generally permissible. However, to assure that the error in reading the frequency is within 0.5 per cent, the oscillator must be correctly calibrated and the scale must be correct. This possible difficulty is avoided by the use of the frequency counter.

The relative error caused in the logarithmic decrement, δ, by incorrect frequency readings is, from Eq 2:

$$\frac{d\delta}{\delta} = \frac{d(f_2 - f_1)}{(f_2 - f_1)} - \frac{df_0}{f_0}$$

Here the difficulty is in the determination of the quantity $(f_2 - f_1)$. The magnitude of this quantity is only about 0.01 of the resonant frequency. Hence an error of 9.5 per cent in reading f_1 and f_2 could cause an error of 100 per cent in the term $(f_2 - f_1)$. By using the frequency counter and taking the average of three readings to determine f_1 and f_2, the maximum error should be less than 2 per cent in logarithmic decrement, δ.

TEST PROCEDURE AND RESULTS

Because of the fundamental nature of this study only one size specimen, the standard 6 by 12-in. cylinder, was used. These were cast in steel molds. The concrete mixes were designed so as to cover almost any strength concrete that might be used on the majority of jobs. The water cement ratio varied in such a manner that the slump varied from approximately 1 to 7 in. Different curing procedures were used and some cylinders were tested wet and others dry. The mixes, curing conditions and results of both the dynamic and static tests are given in Table I. All experimental values represent the average of five cylinders, except in a very few cases where only four were tested.

The fine aggregate was a Wabash River sand with a fineness modulus of about 3.0. The coarse aggregate was a well-rounded Wabash River gravel of 1-in. maximum size. Both of these aggregates passed the usual specification tests. The cement used was a Type I portland cement.

The "double hump" characteristics of the frequency-response curves for certain concrete specimens, as noted by Thomson, were also observed by the authors in only a few of the specimens tested. The following observation was made in testing 6 by 12-in. concrete cylinders. If a double hump was noted, either hump could be made the predominating one by merely changing the plane of vibration. This phenomenon may be caused either by the inherent heterogeneity of concrete

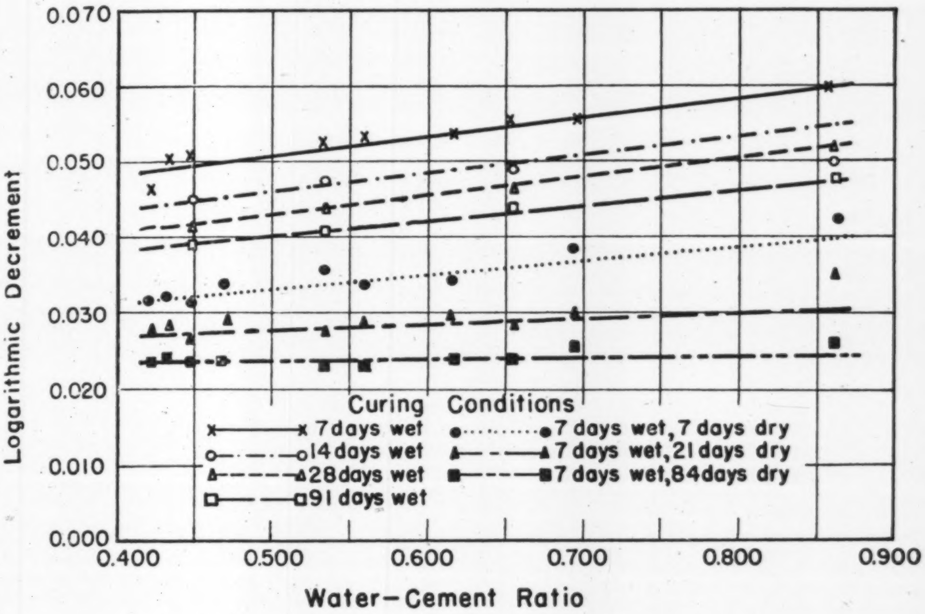

Fig. 3.—Influence of Curing on Logarithmic Decrement.

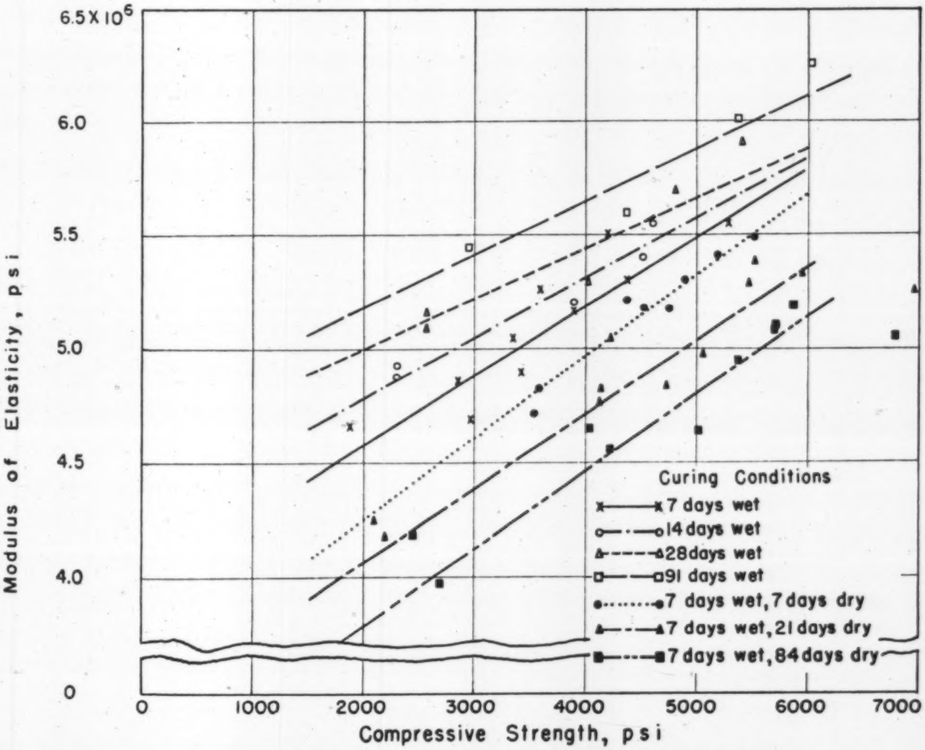

Fig. 4.—Effect of Curing on the Dynamic Modulus of Elasticity.

or by a slight lack of symmetry in the shape of the specimen. As a result, all concrete cylinders tested in this investigation were vibrated in three planes at angles of approximately 120 deg to each other. These readings were averaged and the result was considered to be the reading for the specimen.

A graphical presentation showing the influence of water-cement ratio, age, and curing on the logarithmic decrement is given in Fig. 3. Four conclusions may be drawn from the figure:

1. If the curing conditions are the same, the logarithmic decrement decreases with an increase in age.

2. The logarithmic decrement decreases as the moisture content of the specimen decreases.

3. The logarithmic decrement becomes less dependent on water-cement ratio as the moisture content decreases.

4. Furthermore it may be concluded from the relationship of the logarithmic decrement to the water-cement ratio that logarithmic decrement, if used alone, is not a measure of concrete strength.

The influence of strength, age and curing on the dynamic modulus of elasticity is shown in Fig. 4. The following conclusion may be drawn from this figure:

1. For the same curing conditions, the modulus of elasticity increases as the strength increases.

2. If the concrete is kept moist, the modulus of elasticity increases with age, and if the concrete is allowed to *dry* the modulus of elasticity decreases with age.

3. Modulus of elasticity, if used alone, is not a measure of concrete strength.

The conclusion that neither logarith-

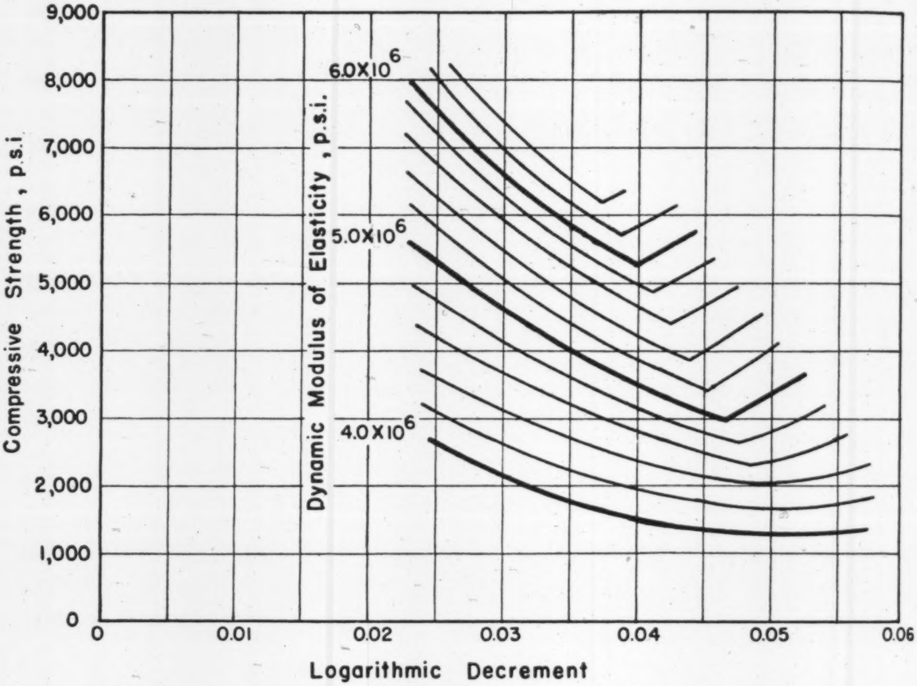

FIG. 5.—Relation Between Dynamic Modulus of Elasticity, Logarithmic Decrement, and Compressive Strength.

mic decrement nor modulus of elasticity alone is sufficient to estimate the compressive strength was predicted on the basis of previous investigations by others. It was further suggested that the combination of logarithmic decrement and modulus of elasticity should be useful in predicting the compressive strength. The interrelation of these three variables has been determined from the test results given in Table I and is represented by the empirically derived curves in Fig. 5.

In general the lines of equal modulus of elasticity which have positive slope indicate concretes that have been moist cured until tested in a wet condition; those with negative slopes indicate concrete that has been allowed to dry, after an initial moist curing period, and tested dry.

With the aid of Fig. 5 it is a simple matter to predict the compressive strength of concrete. The diagram should be entered on the horizontal axis with the logarithmic decrement and this point projected upward to the proper value of the modulus of elasticity; the compressive strength can then be read on the vertical axis. The concrete strength determined in this manner from Fig. 5 will usually agree with the values given in Table I within 10 per cent regardless of age, mix, or curing. The concrete was in all cases a workable mix. Additional experience at the University of Illinois with cylinders whose mix, age, and curing were unknown to the authors indicated that compressive strength could be predicted with an average error of about 3 per cent.

To estimate the strength of concrete using other aggregates such as light weight aggregates, other types of cement and mixes containing admixtures, this diagram may have to be modified.

Acknowledgments:

This work was carried out in Talbot Laboratory as a project of the Engineering Experiment Station in the Department of Theoretical and Applied Mechanics of the University of Illinois.

DISCUSSION

Mr. J. R. Leslie.[1]—Would Mr. Kesler state whether or not he investigated the change in damping factor with frequency or size of the specimen. Presumably the damping factor increases with the frequency of test; hence the smaller the specimen the greater the decrement. Families of curves would be required for different conditions met with in practice.

Mr. C. E. Kesler (*author*).—What Mr. Leslie says is true; as the frequency increases the damping factor increases and the dynamic modulus of elasticity decreases. While not reported in this paper, we have made tests on specimens of different sizes—the different sizes giving different frequencies. We believe that this phenomenon should properly be termed frequency effect and not "size effect" as previously in the literature. Further work should be done along this line so that there need be no restriction on size of specimen for test purposes in order for a laboratory to obtain reproducible results and in order that several laboratories may obtain comparable results, regardless of size of specimen.

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[1] Research Engineer, The Hydro Electric Power Commission of Ontario, Toronto, Canada.

COMPARISON OF DYNAMIC METHODS OF TESTING CONCRETES SUBJECTED TO FREEZING AND THAWING[*]

By G. M. Batchelder[1] and D. W. Lewis[2]

Synopsis

The results of resonant frequency and velocity (soniscope) tests on concrete mixes of varying durabilities when subjected to freezing and thawing are discussed. Changes in velocity squared and in modulus of elasticity calculated from resonant frequency are compared For two mixes, the tests included dynamic determinations of Poisson's ratio. Poisson's ratio values so obtained were used to calculate modulus of elasticity values from velocity, and these were compared with the values obtained from resonant frequency tests.

It was concluded that, for the concretes tested, the velocity tests were less sensitive than resonant frequency measurements as a measure of the deterioration of the specimens. No advantage was found in calculating modulus of elasticity values from the velocity measurements; change in the velocity squared was a better measure of deterioration. Differences between the changes in these two quantities were caused by apparent changes in Poisson's ratio during the freezing and thawing of nondurable concrete.

In recent years, dynamic techniques have been widely used in concrete testing, principally in connection with durability studies. The basic theory on which these test methods are based has been known since 1877, when Lord Rayleigh reported the mathematical relationships existing between resonant frequency of vibration of a specimen, the velocity of sound through the material, and its modulus of elasticity (**12**).[3] The modulus of elasticity may be expressed in terms of the specimen dimensions and density and either the resonant frequency of vibration or the velocity of sound.

Dynamic testing techniques are divided into two general methods, one based upon determination of the fundamental resonant frequency of vibration of a specimen and the other upon measurement of the velocity of a compressional wave through the material.

Investigations of the resonant frequency technique by Powers (**11**), Hornibrook (**4**), Thomson (**13**), Obert and Duvall (**9**), and others led to the publication, in 1947, of an ASTM Tentative Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) (C 215 – 47 T).[4] This method has subsequently been revised and enlarged[5] to include determination of the fundamental torsional frequency, permitting calculation of modulus of elasticity, modulus of rigidity, and Poisson's ratio using the equations given by Pickett (**10**). The use of the fundamental longitudinal frequency to determine the modulus of elasticity is described by Obert and Duvall (**9**).

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[*] Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

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[2] Research Engineer, Joint Highway Research Project, Purdue University, Lafayette, Ind.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1064.

[4] 1949 Book of ASTM Standards, Part 3, p. 855.

Correlations of changes in the dynamic modulus of elasticity with the deterioration of concrete subjected to freezing and thawing and with changes in the flexural strength were made by Hornibrook (**4**), Thomson (**13**), Willis and DeReus (**19**), and others. As a result, the test is widely used in durability testing of concrete. The method is restricted to tests on laboratory specimens with uniform cross-sections.

Investigations using velocity measurements, which are not affected by specimen shape and can be used in the field, apparently were started by Long and Kurtz (**6**). Later reports by Long, Kurtz, and Sandenaw (**7**) and West (**14, 15**) gave test results of a similar nature involving measurement of the transit time of a single impact pulse through the concrete between two pickups.

An electronic instrument, known as the soniscope, was developed by the Hydro-Electric Power Commission of Ontario, Toronto, Can., and was described by Leslie and Cheesman (**5**). Repetitive pulses of high-frequency sound are generated by the instrument, and the transmission time of the signals through the concrete is accurately measured. Reports by Cheesman (**3**), Whitehurst (**16, 17, 18**), Batchelder (**2**), Arndt (**1**), and Meyer (**8**) have illustrated some of the uses of the soniscope in concrete testing. The modulus of elasticity can be calculated from the velocity, the density, and Poisson's ratio for the concrete. Since density and Poisson's ratio are usually unknown for concretes in the field, the test results are frequently left in terms of the velocity.

This paper reports the results of laboratory tests to compare the resonant frequency and velocity test techniques for determining the deterioration of laboratory specimens of concrete subjected to freezing and thawing, and to compare the actual moduli of elasticity values computed from the results obtained by the two test methods. Theoretically, the same results should be obtained from tests on the same specimens by the two methods.

Two series of tests were conducted. The first, designated as series A, involved transverse resonant frequency and velocity tests on three concretes (two mixes of each). Comparisons of test results were made on the basis of changes in velocity squared and in the dynamic modulus of elasticity calculated from the transverse frequency during freezing and thawing. In series B, tests were run on two concretes (one mix of each). Longitudinal, transverse, and torsional frequencies were determined and velocities measured in this series. Values of the modulus of rigidity and Poisson's ratio were obtained, and moduli of elasticity were computed from the longitudinal and transverse frequencies and from the velocities.

MATERIALS AND TEST PROCEDURES

Concrete Materials:

All comparisons made of the test results obtained in this study are restricted to tests made on the same specimens. Therefore, detailed information on the materials used has been omitted.

Series A.—Type I cement, laboratory No. 307, and fine aggregate from source No. 79-1, a glacial material with a good field performance record, were used throughout the test series. The coarse aggregate was a glacial gravel, source No. 84-1G, with a poor field performance record. For part of the mixes, this gravel was modified by removing lightweight particles from it. For mixes 43-1 and 43-2, all material having a bulk saturated surface dry specific gravity less than 2.30 was removed, while material lighter than 2.50 was removed for mixes 45-1 and 45-2.

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[5] Tentative Method of Test for Fundamental Transverse and Torsional Frequencies of Concrete Specimens (C 215 – 52 T), 1952 Book of ASTM Standards, Part 3, p. 1072.

Series B.—A different type I cement, laboratory No. 310, and fine aggregate 79-1 were used in this series. A glacial gravel, 79-1G, with a good field performance record, was used as the coarse aggregate.

Concrete Mixes:

The maximum size of aggregate used was 1 in., with the same gradation used for all mixes. The coarse aggregate for series A was vacuum-saturated before mixing; that for series B was immersed for 24 hr. Air entrainment was accomplished by the use of neutralized Vinsol resin in all mixes except mix 1 in series B.

All concrete was mixed in a Lancaster tub-type mixer and placed in 3 by 4 by 16-in. beam molds. Mixing and placing procedures were the same for all mixes. Duplicate mixes were made on different days for series A.

General properties of the concrete mixes used are shown in Table I. Values shown for series A are the averages for the duplicate mixes; for series B, they are for only one mix.

Curing and Freezing-and-Thawing Tests:

All specimens were cured for 28 days completely immersed in water at 70 F, except those from mix 1, series B, which were cured only 21 days. At the end of the curing period, three beams from each mix in series A and eleven from each mix in series B were subjected to alternate freezing and thawing. The cycle consisted of a 16-hr freezing period and an 8-hr thawing period. Freezing was done in air at −18 F in a walk-in freezer; thawing was in running tap water at 55 F. Resonant frequency and velocity tests was made periodically during freezing and thawing. At the end of the testing, beams were broken in flexure.

Dynamic Tests:

The soniscope used for the velocity tests was built in the Joint Highway Research Project laboratories and used by E. A. Whitehurst in his studies of setting time of concrete (**17, 18**). All velocity tests were made through the length of the specimens (16-in. path length).

TABLE I.—CONCRETE MIX PROPERTIES.

Test Series and Mix Designation	Cement Content, sacks per cu yd	Water-Cement Ratio by Weight	Air Content, per cent	Slump, in.	Unit Weight, lb per cu ft
Series A:					
4A-2 & 3	6.21	0.432	5.1	2¾	146.0
43-1 & 2	6.30	0.452	4.8	2	148.1
45-1 & 2	6.20	0.444	6.0	3	147.7
Series B:					
1.	6.08	0.500	1.0	3	152.6
2.	6.18	0.500	4.7	8	147.0

The resonant frequency test equipment included a Jackson Model 655 oscillator connected to a 4-in. radio speaker with an aluminum rod cemented into the voice coil for the driver unit. The pickup used was a Brush "Vibromike" Model VM-1, the output of which was amplified and displayed on a 5-in. cathode ray oscilloscope.

Series A.—During the freezing-and-thawing tests on series A specimens, periodic measurements were made of velocity and transverse resonant frequency on each specimen. Modulus of elasticity values were calculated from the transverse frequencies, using the equations given in ASTM C 215 – 52 T, Tentative Method of Test for Fundamental Transverse and Torsional Frequencies of Concrete Specimens.[5] Poisson's ratio was assumed to have a value of $\frac{1}{6}$.

Series B.—More complete dynamic tests were conducted on the specimens in series B, where longitudinal, transverse, and torsional resonant frequencies and velocities were measured. Modulus of elasticity, modulus of rigidity, and Poisson's ratio values were determined for each test, using the equations given in the Appendix. The general procedure was as follows:

1. The dynamic modulus of elasticity was calculated from the fundamental longitudinal frequency, neglecting the correction for Poisson's ratio which is very small in value (Eq 2).
2. The modulus of rigidity was calculated from the fundamental torsional frequency (Eq 3).
3. Poisson's ratio was calculated using the values of modulus of elasticity and modulus of rigidity determined above (Eq 4). The value of Poisson's ratio thus obtained was used to determine the correction factors used in the equations in steps 4 and 5. (In a few cases, difficulty was encountered in determining the resonant longitudinal frequency, probably because of limited power of the equipment. In these instances, Poisson's ratio was calculated from the transverse modulus of elasticity.)
4. The modulus of elasticity based upon the fundamental transverse frequency (in the direction of the 4-in. dimension of the specimen) was calculated (Eq 5).
5. A dynamic modulus of elasticity value was calculated from the velocity measurement (Eq 8).

DISCUSSION OF TEST RESULTS

Series A:

Since no attempt was made in this series of tests to determine the value of Poisson's ratio, modulus of elasticity values were not calculated from the velocity measurements. Instead, the results of the tests were compared on the basis of the relative changes in dynamic modulus of elasticity (based on transverse frequency) and in velocity squared. As shown in Eq 8 of the Appendix, the square of the velocity is directly proportional to the modulus of

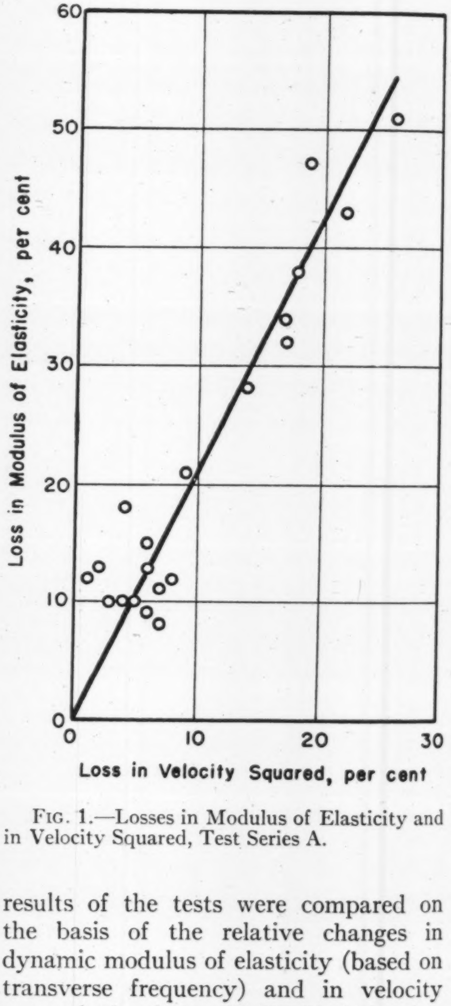

FIG. 1.—Losses in Modulus of Elasticity and in Velocity Squared, Test Series A.

elasticity. Therefore, changes in velocity squared, calculated as percentages of the original value, would be the same as the percentage changes in modulus of elasticity. This assumes that Poisson's ratio, whatever its value may be, remains constant during the freezing-and-thawing cycles.

The results of the tests on series A are shown in Fig. 1, where changes in the transverse modulus of elasticity are plotted against changes in the velocity squared. Each plotted point shows the average values obtained from tests on six specimens, three from each of two mixes, after varying numbers of cycles of freezing and thawing.

The average line for the data shown in Fig. 1 does not indicate the equal changes in modulus of elasticity and in velocity squared that had been expected. Instead, the percentage change in modulus of elasticity is approximately twice as great as the change in velocity squared. The data, then, show that velocity measurements were only about one half as sensitive as transverse resonant frequency tests as a measure of the deterioration of these concretes during freezing and thawing.

Original velocity measurements on these specimens showed a range of values from 14,700 to 15,860 ft per sec. Velocities during the freezing-and-thawing tests were never less than 12,000 ft per sec, although decreases in dynamic modulus of elasticity ranged up to 50 per cent.

No reason was apparent for the lack of correlation of the values obtained in this test series. Among the possible explanations considered were:

1. Poisson's ratio may not remain constant as deterioration progresses. If Poisson's ratio increased with deterioration of the concrete, the effect would be to decrease the modulus of elasticity value calculated from the velocity, thus making changes from the original value greater.

2. The concrete may not be sufficiently homogeneous, especially along the "line path" over which velocity tests are conducted, for the equations relating modulus of elasticity to velocity to be applicable. In this case, correlation of the modulus of elasticity values from resonant frequency and velocity tests would be poor even in initial tests before any weathering cycles were started.

3. The possibility exists that the resonant frequency values are dependent upon the "average" condition of the specimen, while the velocity is measured along a single line. The compressional waves used in the velocity test would tend to travel through the soundest material in the specimen, and the results would not reflect the "average" condition of the concrete.

Series B:

The tests conducted in series B were designed to determine the changes, if any, in Poisson's ratio during freezing and thawing and to compare actual values of the modulus of elasticity calculated from the velocity with the values obtained from the resonant frequencies.

Comparison of Moduli of Elasticity from Longitudinal and Transverse Frequencies.—Only the transverse frequencies are ordinarily obtained in routine dynamic testing. Therefore, it was of interest to compare the modulus of elasticity values so obtained with those calculated from the fundamental longitudinal frequencies. These values for mix 1 are plotted in Fig. 2. Each plotted point represents one test on a single specimen. Excellent correlation is shown, the values lying on or very close to the line of equal values drawn on the graph. Values obtained for mix 2 showed a similarly close correlation.

Comparison of Moduli of Elasticity from Transverse Frequency and Velocity.—In Fig. 3 data from the same specimens in mix 1, showing the relationship of the moduli of elasticity calculated from transverse frequency and velocity, are plotted. As in Fig. 2, the curve shown is the theoretical line of equal values. It is readily apparent that the theoretical relationship does not hold, even though Poisson's ratio was determined for each test and was used in calculating the moduli of elasticity values. There is a definite general trend for the values computed from velocity measurements to be considerably higher than those computed from transverse frequency. It is significant, however, that a somewhat better correlation is shown for the original values—that is, those measured before freezing-and-thawing tests were started. Actually, these original values calculated from velocity tend to be lower than those from transverse frequency. After freezing and thawing, the reverse is true. This shows the same trend previously noted in test series A; that is, the change in modulus of elasticity calculated from velocity undergoes less change as the concrete deteriorates than does the value obtained from resonant frequency.

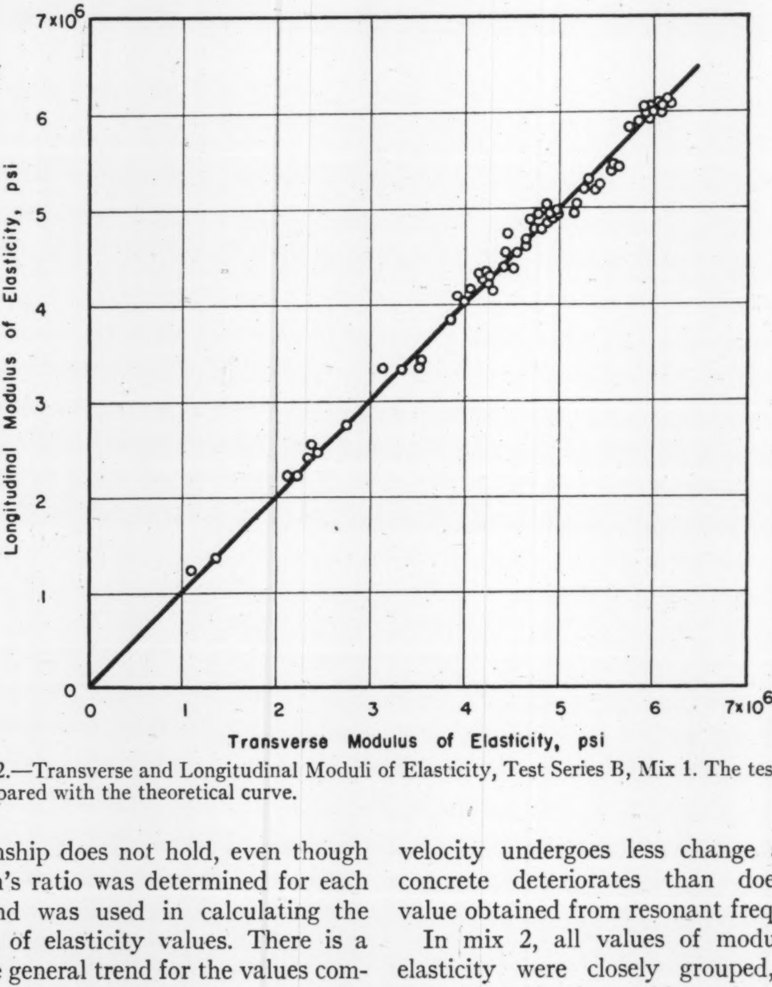

FIG. 2.—Transverse and Longitudinal Moduli of Elasticity, Test Series B, Mix 1. The test values are compared with the theoretical curve.

In mix 2, all values of modulus of elasticity were closely grouped, with little deterioration taking place. The initial values for the eleven specimens are shown in Table II. The values computed from velocity range from 8 per cent higher to 18 per cent lower than those from transverse frequency. Changes in these values were, in general, small during freezing and thawing.

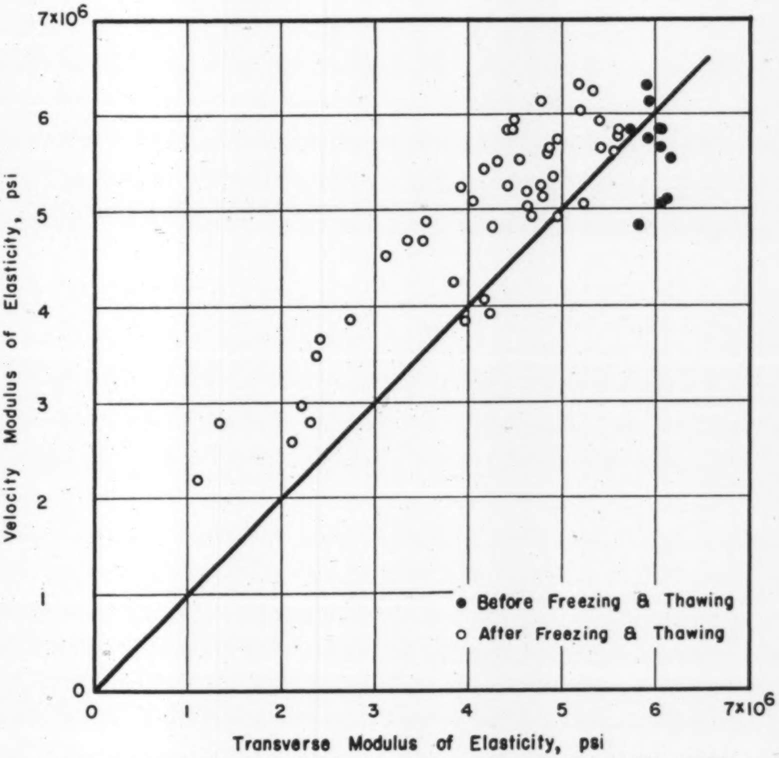

FIG. 3.—Transverse and Velocity Moduli of Elasticity, Test Series B, Mix 1. The test values are compared with the theoretical curve.

The reasonably good correlation noted for the initial values seemed to hold for this concrete mix throughout the tests, probably because of the small changes that took place.

As noted in the Appendix, Long, Kurtz, and Sandenaw (7) list three different equations for converting velocity to dynamic modulus of elasticity, depending upon whether the tests are run on laboratory specimens, pavements, or mass concrete. Leslie and Cheesman (5) recommend that the equation for mass concrete (Eq 8, Appendix) be used for all concrete, including laboratory specimens. This equation was used in this study, and the results confirm their recommendation by indicating reasonable agreement with other dynamic moduli values for concrete that has not undergone serious deterioration. Use of either of the other equations suggested by Long, Kurtz, and Sandenaw would

TABLE II.—INITIAL MODULUS OF ELASTICITY VALUES, SERIES B, MIX 2.

Beam	Modulus of Elasticity, psi		Per cent Difference
	Transverse	Velocity	
No. 1	4.77×10^6	5.16×10^6	+8
No. 2	4.66	4.82	+3
No. 3	4.84	4.20	−13
No. 4	5.04	4.11	−18
No. 5	4.51	4.12	−9
No. 6	4.53	4.22	−7
No. 7	4.65	4.25	−7
No. 8	4.49	4.30	−4
No. 9	4.77	5.16	+8
No. 10	4.93	4.98	+1
No. 11	4.99	4.97	0
Average	4.74	4.57	−3.6

result in much higher values of the velocity modulus of elasticity.

Changes in Dynamic Moduli Calculated from Resonant Frequencies.—Some of the specimens in both mixes of series B were removed from freezing and thawing and broken in flexure or subjected to other tests after only a few cycles had been run on them. As a result, only two specimens from each mix (specimens 1 and 2 in each case) completed the maximum number of cycles of freezing and thawing—36 cycles for mix 1 and 50 cycles for mix 2. The results obtained on different specimens from the same mix agreed quite closely; therefore, the results shown in this section are those for the specimens that completed the greatest number of cycles of weathering.

TABLE III.—DYNAMIC TEST VALUES DURING FREEZING AND THAWING, SERIES B, SPECIMENS 1 AND 2, MIX 1.

Number of Cycles of Freezing and Thawing	Dynamic Moduli and Velocity Squared, per cent of Original Value					Poisson's Ratio
	Longitudinal Modulus of Elasticity	Transverse Modulus of Elasticity	Modulus of Rigidity	Velocity Squared	Velocity Modulus of Elasticity	
0	100	100	100	100	100	0.30
1	88	90	90	96	102	0.27
2	82	82	85	94	106	0.24
4	76	75	80	82	94	0.24
6	68	70	72	79	90	0.24
10	56	57	58	70	82	0.23
15	44	42	45	56	63	0.25
20	40	39	42	48	55	0.23
25	36	36	39	41	48	0.23
36	21	20	24	33	43	0.15

Tables III and IV show the changes in dynamic moduli, velocity squared, and Poisson's ratio during freezing and thawing for mixes 1 and 2, respectively. Except for Poisson's ratio, the values are shown as percentages of the original value at the end of the curing period.

The data for both mixes show a very close correlation in the changes in the values of the dynamic moduli of elasticity calculated from longitudinal and transverse frequencies and in the values of modulus of rigidity. These three test values, on the basis of percentage of original value, are practically identical throughout the tests. This indicates that measurements of longitudinal, transverse, and torsional frequencies are equally useful as a means of measuring the deterioration of concrete specimens subjected to freezing and thawing. For practical purposes, they are equally sensitive in detecting such deterioration. Poisson's ratio does not affect the modulus of rigidity; and the variations in Poisson's ratio encountered in this study caused maximum changes of 1.5 and 0.5 per cent in the modulus of elasticity calculated from transverse and longitudinal frequencies, respectively. Therefore, with any specimens whose weight changes are negligible during weathering, as was the case in these tests, the squares of the frequencies could be used as the measure of the deterioration, without serious loss in accuracy. Thus, all calculations of dynamic moduli would be eliminated.

TABLE IV.—DYNAMIC TEST VALUES DURING FREEZING AND THAWING, SERIES B, SPECIMENS 1 AND 2, MIX 2.

Number of Cycles of Freezing and Thawing	Dynamic Moduli and Velocity Squared, per cent of Original Value					Poisson's Ratio
	Longitudinal Modulus of Elasticity	Transverse Modulus of Elasticity	Modulus of Rigidity	Velocity Squared	Velocity Modulus of Elasticity	
0	100	100	100	100	100	0.29
1	94	92	94	96	92	0.30
2	92	92	93	98	95	0.29
4	93	93	94	97	96	0.28
6	91	92	93	95	94	0.28
10	91	91	91	95	97	0.27
15	92	92	92	91	91	0.28
20	93	92	92	92	90	0.29
25	94	92	92	90	86	0.30
35	95	93	92	91	86	0.30
50	94	93	92	87	85	0.31

Changes in Velocity Squared and Dynamic Modulus of Elasticity Calculated from Velocity.—The changes in velocity squared and in modulus of elasticity calculated from velocity, shown in Table III, do not correspond well with the changes in the other dynamic moduli. The tendency, although not so pronounced, is the same as that encountered in test series A. The losses in velocity squared are less than the losses in the moduli of elasticity calculated from resonant frequencies (a greater percentage of the original value is retained).

Mix 2 data, shown in Table IV, indicate a much closer correlation of changes in the velocity values with changes in the other dynamic moduli. Apparently the closeness of the correlation is affected by the amount of deterioration that takes place. In the case of mix 2, there is little choice between velocity squared and modulus of elasticity calculated from velocity. Velocity squared does, however, tend to be a little closer to the other values after 15 cycles of freezing and thawing.

The results obtained in these tests indicate no benefits from determining Poisson's ratio and calculating values of modulus of elasticity from velocity measurements. Instead, the velocity values themselves appear to be a better measure of concrete deterioration than do the modulus of elasticity values calculated from velocity. These results confirm the opinion expressed by Whitehurst (17, p. 1182) that velocity measurements should be used as such, without attempting to calculate the dynamic modulus of elasticity.

Actual velocity values obtained for the specimens in test series B (other data shown in Tables III and IV) varied from 15,470 ft per sec initially to 9920 ft per sec after weathering for mix 1; and from 14,200 to 13,350 ft per sec for mix 2.

Changes in Poisson's ratio.—The data in Table III show a definite trend in the values of Poisson's ratio, which decreased markedly as deterioration of the concrete took place. It should be noted that the change is in the opposite direction from that required to improve the correlation of the dynamic moduli changes from resonant frequency and velocity measurements. The effect of the variation in Poisson's ratio is to cause differences in the relative changes in velocity squared and in modulus of elasticity calculated from velocity. The changes in Poisson's ratio cannot account for the discrepancies in results noted above in the series A tests.

The changes in the calculated values of Poisson's ratio are caused by the difference between the changes in longitudinal modulus of elasticity and in modulus of rigidity. Although the percentage difference in the changes in these values is quite small, it is consistent. The change in modulus of rigidity is slightly less than the change in modulus of elasticity, resulting in an apparent decrease in Poisson's ratio. Although the significance of this change is difficult to determine, it seems reasonable that actual measurements of Poisson's ratio would be superior to the use of assumed values for calculation of modulus of elasticity from velocity determinations. Use of the measured values, however, causes greater discrepancies between changes in the velocity and resonant frequency moduli than does the assumption of a constant Poisson's ratio during the weathering tests.

The values of Poisson's ratio for mix 2 (Table IV) remained relatively constant during the freezing-and-thawing cycles. It appears probable that this is due to the small amount of deterioration that took place in this concrete mix. Since no great changes took place in the other characteristics of the concrete, Poisson's ratio might be expected to undergo but little change.

CONCLUSIONS

On the basis of the test results reported and discussed above, the following conclusions are believed to be justified:

1. In general, no benefit is derived from calculating modulus of elasticity values from velocity measurements. When the concrete undergoes extensive deterioration, the changes in velocity squared form a more accurate indication of concrete deterioration than does such a modulus of elasticity.

2. If the modulus of elasticity is calculated from velocity, the equation recommended by Leslie and Cheesman should be used (Eq 8, Appendix). Although inaccurate for the deteriorated concrete tested, this equation results in better correlation with resonant frequency moduli than do the other equations that have been suggested for velocity modulus of elasticity.

3. Velocity measurements are less sensitive to deterioration than are resonant frequency determinations. Decreases in resonant frequency moduli may be twice as great as the decreases in velocity moduli as the concrete deteriorates.

4. Measurements of longitudinal, transverse, or torsional resonant frequencies are equally useful and sensitive in tracing the deterioration of concrete specimens.

5. Poisson's ratio showed a definite decrease in the nondurable concrete, as the weathering cycles progressed. This change is responsible for the differences noted between changes in velocity squared and in the modulus of elasticity calculated from the velocity.

6. It appears probable that the lack of correlation between changes in resonant frequency and velocity moduli, when the concrete deteriorates, is due to failure of the velocity measurements to indicate the "average" condition of the specimen. The pulse path in the velocity measurements would probably be through the soundest concrete in the interior of the specimen, and the results would indicate only the condition of the best portion of the concrete. An "average" condition for the entire mass of concrete should be indicated by the resonant frequency tests in which the whole specimen is vibrated.

APPENDIX

EQUATIONS FOR CALCULATION OF DYNAMIC MODULI AND POISSON'S RATIO

Modulus of Elasticity from Longitudinal Resonant Frequency:

Obert and Duvall (9) give the equation relating longitudinal frequency to modulus of elasticity as follows:

$$E = \frac{4L^2\rho n^2}{i^2\left(1 - \dfrac{i^2\pi^2\mu^2K^2}{L^2}\right)} \tag{1}$$

where:

E = modulus of elasticity,
L = length of specimen,
ρ = density of specimen = weight divided by gravitational acceleration,
n = longitudinal resonant frequency,
i = integer depending upon the tone of vibration ($i = 1$ for fundamental tone),
μ = Poisson's ratio, and
K = radius of gyration of section normal to axis.

The correction factor involving Poisson's ratio is quite small and may be neglected without serious error. Eliminating this factor the equation becomes:

$$E = 4L^2\rho n^2 \tag{2}$$

for the fundamental longitudinal resonant frequency. The maximum variation in E that would have been introduced by the correction for Poisson's ratio was less than 0.5 per cent in this study.

Modulus of Rigidity:

The equation given by Pickett (10) and in ASTM Method C 215[5] was used to calculate modulus of rigidity values:

$$G = BWn^2 \tag{3}$$

with $B = \dfrac{4LR}{gAi^2}$

and $R = \dfrac{a/b + b/a}{4(a/b) - 2.52(a/b)^2 + 0.21(a/b)^6}$

where:
G = shear modulus or modulus of rigidity,
W = weight of specimen,
n = resonant torsional frequency,
L = length of specimen,
g = gravitational acceleration,
A = cross-sectional area of specimen,
i = integer equal to mode of vibration (1 for fundamental mode),
a = length of shorter side of rectangular specimen, and
b = length of the longer side of the cross section.

Poisson's Ratio:

The equation for Poisson's ratio (10)[5] is:

$$\mu = \frac{E}{2G} - 1 \tag{4}$$

where:
μ = Poisson's ratio,
E = modulus of elasticity, and
G = modulus of rigidity.

Modulus of Elasticity from Transverse Resonant Frequency:

The equation used to calculate the modulus of elasticity from transverse resonant frequency is as follows (10)[5]:

$$E = CWn^2 \tag{5}$$

with $C = 0.00245\,\dfrac{L^3T'}{bt^3}$ and

$$T' = T\left(\frac{1 + (0.26\mu + 3.22\mu^2)K/L}{1 + 0.1328K/L}\right)$$

where:

E = modulus of elasticity, psi,

W = weight of specimen, lb,

n = fundamental transverse resonant frequency, cps,

L = length of specimen, in.,

t, b = dimensions of prism cross-section, t being in the direction of vibration, in.,

T' = correction factor dependent on Poisson's ratio and the ratio K/L,

T = correction factor for Poisson's ratio of $\frac{1}{6}$ (from tabulated values given in references above),

K = radius of gyration of the prism, and

μ = Poisson's ratio.

Modulus of Elasticity from Velocity:

Long, Kurtz, and Sandenaw (**7**) suggest the use of the following equations relating velocity of sound through concrete with the modulus of elasticity:

For laboratory specimens:

$$E = V^2\rho \qquad (6)$$

For pavements:

$$E = V^2\rho(1 - \mu^2) \qquad (7)$$

For mass concrete:

$$E = V^2\rho\,\frac{(1 + \mu)(1 - 2\mu)}{(1 - \mu)} \qquad (8)$$

where:

E = modulus of elasticity,

V = compressional wave velocity,

ρ = density of the concrete, and

μ = Poisson's ratio.

Leslie and Cheesman (**5**) recommend the use of Eq 8 for all concrete. It was used for the calculations in this study.

It should be noted that the three equations would give greatly differing values for the modulus of elasticity with given velocity, density, and Poisson's ratio values. For example, if Poisson's ratio were 0.30, E values equal to $V^2\rho$, 0.91 $V^2\rho$, and 0.743 $V^2\rho$ would be obtained by use of Eqs 6, 7, and 8, respectively.

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DISCUSSION

MR. E. A. WHITEHURST.[1]—The authors are to be commended for undertaking a study which has long been needed. It is to be hoped that future studies of this nature will clearly determine the true meaning of the various dynamic testing techniques which may now be applied in concrete testing. The need for such determination has become increasingly evident during the past few years.

The conclusion of the authors that the measurement of longitudinal, transverse, or torsional resonant frequencies are equally useful and sensitive in studying the deterioration of concrete specimens is a valuable one, in that certain sizes and shapes of specimens lend themselves better to one of these techniques than to others. The authors' finding concerning variations in Poisson's ratio during progressive weathering cycles is also valuable. This finding, which apparently has not been previously reported, may shed light on many of the previously unexplainable phenomena observed in concrete testing.

The writer is in complete agreement that, in general, no benefit is derived from calculating the modulus of elasticity from the values of velocity measured through the concrete. It is also agreed that the equation recommended by Leslie and Cheesman, that is, that developed for the relationship between dynamic modulus and pulse velocity through mass concrete, should be used if such calculation is to be undertaken.

The writer wishes to differ slightly with the authors in their conception of the resonant frequency technique as indicating the "average condition" of the specimen and the pulse velocity technique as indicating some other value. Those who have made use of velocity techniques during the past few years have been prone to state that this technique resulted in measurement of the average quality of concrete along a line through the tested material. This is very probably true in the case of tests on structures where relatively long path lengths are involved. It would seem probable, however, that in the case of small laboratory specimens neither the velocity technique nor the resonant frequency technique measures the true average character of the concrete. The pulse undoubtedly follows a path near the center of the specimen where deterioration has been least and velocity is highest. It might be supposed, therefore, that the velocity technique measures the "best average" condition of the specimen. Although there is little evidence to indicate exactly to what degree the resonant frequency of the specimen is influenced by spalling and cracking, the fact that at advanced stages of deterioration peculiar multiple peak resonance curves are frequently found and that in some cases the specimen cannot be driven in its fundamental mode at all would indicate that cracking and spalling do have some influence upon the resonant frequency. It might then be supposed that the resonant frequency technique measures the "worst average" condition of the specimen. It may be argued, then, that neither technique results in a true measure of the quality of the concrete and that this true condition lies somewhere between the two.

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The writer submits that, if the above argument may be admitted, it becomes important to consider not only the results of these tests upon laboratory specimens but the proposed use to which the concrete is to be put. If the concrete is to be used in thin slabs where flexural strength is highly important, it is suggested that the resonant frequency technique results should be heavily considered. Although these results indicate the worst conditions of the laboratory specimens, it is probable that this condition may predominate in the slabs. If, on the other hand, the concrete is to be used in more massive members, it seems highly possible to the writer that the results of resonant frequency tests may be overly critical and may imply a low degree of durability which will not exist in the field.

Dynamic testing techniques, particularly those involving the measurement of resonant frequency, have proved extremely useful in the laboratory for a number of years. Because of the facility with which such tests may be made, they have been widely accepted. It is only during the past few years that much attention has been paid to dynamic testing techniques involving the measurement of pulse velocities through concrete. Since these may be made with equal facility on field structures, there is certain to be a strong demand for continued development of such techniques and apparatus. As it has been clearly shown that the two approaches do not always yield the same results, further studies are indicated to show just what the relationship between the two tests is and what relationship they bear to other properties of the concrete. The writer urges, however, that in making such evaluations and comparisons the results of resonant frequency tests not be considered as necessarily correct and the results of tests of other nature considered incorrect insofar as any difference between the two occurs. It should always be remembered that we do not really know very much about the true meaning of either of these tests and that the likelihood of error in evaluating the results is about equally great for both.

It is to be hoped that further studies of the nature reported in this paper will be undertaken in the near future and that these will contribute to an early and clear understanding of the meaning of and limitations of various dynamic testing techniques which have been developed during the past few years.

MR. BRYANT MATHER.[2]—The authors, and other workers in the field of pulse velocity testing, characteristically state the presumed mathematical relation between velocity and modulus of elasticity; then they characteristically advise one not to engage in those mathematics. The question I would like to raise is this: Is there really any greater validity in dealing with values of modulus of elasticity calculated from resonant frequency, or perhaps is it merely that the resonant frequency methods came first and we got in the habit of calculating modulus of elasticity from them? In the authors' figures dealing with resonant frequency data all values were expressed either in actual or relative modulus of elasticity. This is customary. Perhaps it would be better to deal in values of resonant frequency squared even as it is suggested that we should deal in values of velocity squared.

MR. STANTON WALKER.[3]—One reason for computing the modulus of elasticity instead of only using the square of the frequencies is to permit comparisons of results secured from specimens differing in dimensions. It is gratifying to know that, for routine work in the laboratory using standard sizes of specimens, the simple measurement of fundamental frequency appears to give results as satisfactory as more complex dynamic methods.

MESSRS. J. R. LESLIE[4] AND W. E. PARKER.[4]—The authors' paper has been read with great interest, particularly because some of their results appear to be partly at variance with the results of similar tests carried out in the Research Division of the Hydro Electric Power Commission of Ontario.

It should be agreed by all concerned in the problem of dynamic testing that numerical values obtained in one method of test need not agree closely with those obtained in a different way. Both methods may of course provide useful information. This is the case when comparing sonic and pulse tests because, as the authors point out, the sonic modulus is the average modulus of the specimen. It takes into account deterioration and cracking at the surface as well as in the center of the cylinder. The pulse method involves the concrete in the most direct line joining the transducers. To illustrate the differences, a small saw cut may be started part way through the test specimen without affecting the pulse velocity; however, a change of 20 per cent or more in sonic modulus results.

Similar reasoning could explain observed differences in sonic and pulse results, where, despite visible deterioration, concrete of good quality remains in the interior. Differences in the two methods may be, in themselves, used to study the characteristics of the internal material.

Pulse velocity tests frequently yield different results when deteriorated concrete specimens are tested along different axes. The difference observed serves in a general way to indicate the orientation of internal lamellar cracking.

Pulse tests may be carried out, in some instances, on specimens in an advanced stage of deterioration, when resonant methods fail.

The change of Poisson's ratio with deterioration is an interesting effect not noted in the literature to date. The disagreements noted heretofore in the published values for Poisson's ratio may now be explained.

MESSRS. G. M. BATCHELDER AND D. W. LEWIS (*authors' closure*).—The discussions by Mr. Whitehurst and Messrs. Leslie and Parker emphasize that both resonant frequency and velocity techniques can provide valuable information even though the numerical results may differ. The choice of technique to be used should depend upon the purpose of the tests and the test conditions.

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[3] Director of Engineering, National Sand and Gravel Assn., and National Ready Mixed Concrete Assn., Washington, D. C.

[4] Research Engineers, Hydro Electric Power Commission of Ontario, Toronto, Canada.

The tests described in the paper were conducted to determine the relative durability of concretes for pavements, where flexural strength changes caused by freezing and thawing are important. For this purpose the resonant frequency techniques are believed to be the best, and the changes in modulus of elasticity can be correlated with the changes in flexural strength. For other purposes, pulse velocity tests will yield information that cannot be obtained by resonant frequency methods. These include field tests of actual structures, laboratory tests on specimens unsuitable for resonant frequency tests because of their size, shape, or severe state of deterioration, and tests to determine differences in quality in various parts of a structure or specimen.

With regard to Mr. Mather's question, it would appear more valid to calculate modulus of elasticity from frequency than from velocity, since incorrect values of Poisson's ratio have a much smaller effect upon the results obtained from resonant frequencies. For the comparison of test results on different sizes of specimens mentioned by Mr. Walker or the comparison of the results of longitudinal and flexural frequency tests shown in the paper, modulus of elasticity calculations are necessary. It is true, however, that resonant frequencies can usually be employed in the same manner as velocities. Where weight changes of the specimens are negligible during test and changes are to be stated in relative or percentage values, use of the frequency squared will give the same results as will use of the modulus of elasticity values.

EVALUATION OF CURING COMPOUNDS FOR PORTLAND CEMENT CONCRETE*

BY C. E. PROUDLEY[1]

SYNOPSIS

Desirable characteristics of curing compounds for use on highways and structures are enumerated and laboratory tests simulating field conditions for the purpose of evaluating liquid, membrane-forming compounds are described. Factors affecting the correlation between laboratory tests and service performance are discussed as are the difficulties encountered in securing uniformity among testing laboratories under the present methods. Influence of job operations and climatic conditions upon the performance of curing compounds is touched upon briefly. Some opinions are expressed regarding the need for practical studies of relative merits of different methods of curing concrete.

Methods used for curing portland-cement concrete are intended to develop the desirable characteristics of the concrete within a specified time limit. A committee of the Highway Research Board defines curing as "... the process of maintaining a satisfactory moisture content and a favorable temperature in concrete during the period immediately following placement so that hydration of the cement may continue until the desired properties are developed to a sufficient degree to meet the requirements of service" (**1**).[2] Sometimes the conditions provided artificially by the curing method prevail naturally and the application of curing is superfluous; however, in order that the conditions for curing may be reasonably uniform throughout construction, the application of some kind of curing is invariably specified.

For the past ten years or more curing compounds that are sprayed on the fresh concrete have been favored by many construction agencies and particularly by the contractors who are saved rehandling of cover materials and the hauling or piping of water. Economies effected by a curing method constitute part of the economy of concrete construction. A curing compound retards the escape of mixing water by forming a membrane seal, and after it is applied, no further attention is required. In order to provide satisfactory curing, the membrane must be relatively impermeable for a reasonable curing period, must not affect the quality of the concrete adversely, and should minimize internal temperature fluctuations caused by atmospheric temperatures and radiant heat or, at least, should not exaggerate temperature variations.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Chief Materials Engineer, North Carolina State Highway and Public Works Commission, Raleigh, N. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1077.

This paper will consider nonbituminous liquid compounds only, although asphaltic emulsions and some types of cutbacks have been used successfully, especially when covered with a coating of whitewash to reduce heat absorption. In passing it should be mentioned that the bituminous material is not readily removed when its function as a curing agent is finished. In some cases, however, this is not objectionable as, for example, when the concrete is a tunnel lining or a base for bituminous pavement.

DESIRABLE CHARACTERISTICS

The features desirable in curing compounds are that they be:

1. Impermeable.
2. Noninjurious to concrete.
3. Reflective to radiant heat.
4. Sprayable.
5. Easily removed from concrete after curing period.
6. Quick-drying.
7. Safe both as to flammability and toxicity.

Impermeability.—Although impermeability is the characteristic most emphasized, others are essential if efficient curing is to be obtained. For example, it is desirable to apply the compound to the concrete at the proper time after finishing operations as recommended by Swayze (2) if the most wear-resistant surface is to be secured. If applied too early and the concrete bleeds, the film will not adhere and may be broken by movement of the water under the film. If applied too late, there may be less than the optimum quantity of water present for hydration and, also, the film may not be so impervious to the passage of water due to its having a rougher and more absorbent surface to cover and seal. Between these two extremes the performance of the compounds currently marketed will vary in efficiency. Some formulations are superior when applied to a wet surface while others are preferred if the concrete is to be coated after becoming relatively dry.

Noninjury to Concrete.—Some resins and waxes or other ingredients of liquid curing compounds act on portland-cement mixtures to retard or completely inhibit hydration. Occasionally a curing compound will be encountered which contains such substances. The effects will extend into the concrete a relatively slight distance, but rapid and usually irregular wear of the surface will result. Because such deleterious effects are definitely objectionable, reference is made to the possible reaction of the compound with the concrete in practically all specifications. Some kind of hardness test must be described in the specifications since there is no standard to which one can refer.

Heat Reflectance.—Protection from temperature changes has been proved important in minimizing undesirable cracking of concrete pavements. Blankets of earth, fabric, paper, etc. give protection against extremes of heat and cold whereas compounds of the so-called clear type may even exaggerate temperature variations. For this reason compounds have been developed which contain pigments for the purpose of reflecting heat radiated from the sun. Pigmented compounds are either white or gray.

Sprayable.—Sprayability at temperatures of construction is an obvious necessity. This means that the compound should remain quite fluid while being applied. Most specifications contain a clause pertaining to application of compounds on vertical surfaces. The compounds must not run or sag and, in this respect, as in most others, they are similar to paints and varnishes.

Removability After Curing.—Criticism has been made, especially of the pigmented compounds, that they cause the surface of pavement to be slippery, a serious objection in the case of highways. This slipperiness may be the result of applying more than the specified amount of compound. If the compound deteriorates at the proper rate, it can be removed by brooming or scouring, thereby eliminating this hazard to traffic.

Complaints have been voiced that curing compounds prevent the adhesion of joint fillers and traffic paint to the concrete surface. This is true if filler material and paint are applied before the compound has deteriorated as it is supposed to do. The disintegration of the film will take from several weeks to about a year if exposed to the elements and longer if the surface is protected from the weather. It is advisable, therefore, to remove the curing membrane from surfaces to be given any subsequent treatment. Since this may be necessary, the membrane should be readily removable by brushing with a stiff brush or broom after the specified curing period is completed. A statement of the time for disintegration of the curing film is a proper part of specifications for curing compounds.

Quick-Drying Properties.—The compound should be quick-drying or, rather, should dry to touch in about the same time that is required for the concrete to attain its set. The film should be flexible so long as the concrete may change in volume or shape due to setting, but thereafter it should attain sufficient durability to resist rain, damage by foreign matter blowing on it, or other accidents which might puncture it.

Flammability and Toxicity.—Most of the compounds in use today contain 60 per cent or more of a flammable volatile solvent. It might be thought that such a material would be a fire hazard, but no instance of a flash fire resulting from its use has been reported. Millions of gallons of asphaltic road materials containing high percentages of similar solvents are used each year with only rare accidents resulting from explosions or fire. In view of such experience the specification that the flash point of a curing compound shall be not less than 80 F, or a somewhat higher figure if tested by the open-cup method, seems relatively unimportant. Similarly, the commonly used solvents are injurious to health only if carelessly inhaled in concentrated doses or for long-continued periods. Mention in specifications of flammability and toxicity is a proper precautionary measure, but they are characteristics that will seldom be used as a cause for rejection unless the compound is to be used in a poorly ventilated space.

LABORATORY TESTS

The development of standardized tests for evaluating efficiency of methods of curing concrete has been slow. Most attention has been given to the water retention or properties of impermeability of compounds, since it is obvious that any curing method or material must assure the presence of water for hydration of the cement. ASTM Method C 156[3] has been published as tentative since 1940 with a number of major revisions and many minor ones. Attempts have been made to devise a simpler method but without success. The factor which introduces the greatest complication to the method is the use of portland-cement mortar for a test specimen to which the curing compound under test is applied. No standard portland cement has been made available to combine with distilled water and standard Ottawa sand to yield a standard mortar for the test specimen; therefore, the necessary conditions for a satisfactorily reproducible and reliable test of the curing material are not established. It is known that characteristics of the portland cement and also of the aggregate in mortar or concrete affect the behavior of the curing compounds, and for this reason it is essential that the test include the application of the compound to a portland-cement mixture to correlate laboratory determinations with actual practice.

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[3] Tentative Method of Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156 – 52 T), 1952 Book of ASTM Standards, Part 3, p. 1126.

Other methods for measuring the water retention of films formed by curing compounds have been used which are variations of the ASTM Method C 156. One of the more radical variations combines determination of water loss with absorption of radiant heat (3). Instead of placing the test specimens in a cabinet at 100 F and 32 ± 2 per cent relative humidity for 72 hr, as is prescribed in Method C 156, the specimens are placed under flood lamps so adjusted that the temperature at the surface of an untreated, dry specimen is 120 F. The surrounding air temperature and humidity are those of the laboratory, and a moderately strong draft from a fan keeps the air moving. The duration of exposure is 5 hr.

A number of methods of detecting the possible adverse effects that curing compounds may have on portland-cement concrete have been devised and used. Sand blast, shot blast, and other abrasion test methods are indicative within limits but these require relatively elaborate apparatus; in addition, the results must be interpreted by an experienced concrete technician. Almost as satisfactory is the comparison of the treated and untreated concrete by scratching with a hard metal tool such as a knife or screwdriver. Allowance must be made for the condition of the untreated specimen—that is, the specimen that has been cured with water instead of the compound—since it will be found that at early ages the water-cured specimen may seem to be harder even though the compound has not been injurious to the concrete. Such a method of test depends entirely upon the operator's judgment which is always subject to question. Nevertheless, it serves as a preliminary or "screening" observation and, in case of doubt, further study by other operators or by use of more elaborate test procedure would be conducted to verify the operator's opinion.

The comparatively recent development in curing compounds of the use of opaque pigments to give reflectance to infrared rays and thereby reduce temperature and volume change has necessitated tests of heat reflectance of the surface so cured. Specifications for pigmented curing compounds include a requirement for apparent daylight reflectance which may be determined by means of ASTM Standard Method of Test for Daylight 45-deg, 0-deg Luminous Directional Reflectance of Paint Finishes.[4] This compares the reflectance of the surface coated with the pigmented compound with a surface coated with magnesium oxide smoke. Only a minimum reflectance is specified, usually around 50 per cent.

No test for spraying characteristics has been developed beyond a description of the character of the film produced. Whether it will be sprayable at 35 F or at some other temperature can be determined readily by refrigerating a spray gun containing the compound and observing its behavior when operated at a temperature of application which will prevail in the field. Specifications require that the film be continuous and free from pinholes and such imperfections as might occur if the sprayability of the liquid compound was unsatisfactory. In addition, it must not run or sag when applied at the specified rate. No standards have been suggested, but there should be little difference of opinion among the observers who accept or reject on the basis of sprayability.

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[4] Standard Method of Test for Daylight 45-deg, 0-deg Luminous Directional Reflectance of Paint Finishes (D 771 - 47), 1952 Book of ASTM Standards, Part 4, p. 550.

Drying time for curing compounds is determined in the same manner as for paint, varnish, and lacquer except that the curing compound is applied to a mortar or concrete specimen. The dry-to-touch time is most often specified, and requirements vary from 30 min to 4 hr. Sometimes it is required that the film dry hard within some period between 4 and 12 hr.

When the test for water retention efficiency was first developed, the size and shape of the specimens were chosen to permit making beam tests for strength. Tests reported by Jackson and Kellermann (4) on various methods of curing included strength tests, but these were intended to be a measure of the relative effectiveness of the methods rather than an indication of injurious effects although it could be a combination of the two. Beam strength tests are seldom, if ever, specified as an acceptance requirement for curing compounds.

The ease with which the film can be removed could be a subject for much controversy since it involves the amount of work which must be done to effect its removal and, also, what constitutes removal. Here again there is no standardized test and it is assumed that each purchaser will determine by trial under simulated field conditions whether or not the membrane can be removed to his satisfaction with a reasonable amount of effort after some certain period of time.

Most colorless or pigmented compounds use a solvent which is flammable and, although the record of explosion or fire leads us to believe that the danger is practically nil when handled with ordinary care, some specifications include a flash point requirement. The Interstate Commerce Commission imposes certain restrictions (5) on packaging and shipping materials which have a flash point of less than 80 F by the Tag Open-Cup Tester, and this is approximately the value used when specified.

There is some toxicity associated with practically all volatiles that might be included in the formulation of a concrete curing compound; however, only those which are dangerously toxic are ruled out, such as carbon tetrachloride, benzol, toluene, and methyl alcohol. It may be assumed that the presence in the compound of any substance regarded by physiologists as toxic to human beings is ample justification for rejecting it.

SIMULATING FIELD CONDITIONS IN THE LABORATORY

Field conditions under which concrete is to be cured vary between wide limits depending primarily on climatic conditions. Add to this the possible variations in construction procedure and it is apparent that no one set of laboratory conditions may be used to give a comprehensive evaluation of a curing material or method. As is usual, some compromise is desirable for the sake of economy of time and effort. A condition of average severity would seem reasonable, and there has been little argument against 100 F and a relative humidity of 30 to 34 per cent as the average field conditions to be simulated in laboratory tests for moisture retention.

Temperatures at which use of curing compounds may be permitted range from the lowest at which concreting operations are allowed up to the hottest summer day in a southern state This atmospheric temperature range is from about 35 F, below which insulation or artificial heat would almost certainly be applied, up to about 110 F which would not exist for more than an hour or two during the day. The internal temperature of the concrete will generally be 5 to 15 F higher at both the cold and hot extremes.

The humidity varies throughout the day as the atmospheric temperature changes. The need for protecting the concrete from loss of moisture is less as the relative humidity increases. When the temperature of the concrete is higher than the dew point of the surrounding atmosphere, there is a "boiling" effect; this is particularly noticeable when there is radiant heat from the sun Under extreme conditions blisters may be formed in the freshly applied curing compound unless the manufacturer has used a formulation to avoid them. Blisters are an obvious point of weakness in the curing membrane and for this reason a radiant-heat test is desirable as previously mentioned (3).

Construction procedures complicate the effort to simulate field conditions. Not only do the differences among cements and aggregates have an effect upon the behavior of the compounds but the methods used by the constructor will play an important part. Chief of these is the time at which the compound is applied to the finished structure. A troweled finish will give a different effect from that of a drag or broom finish just as an aggregate with a high percentage of fines below the No. 50 and No. 100 sieve sizes will result in a smoother surface. The richness of the mixture or its wetness will affect the surface texture and, consequently, the results obtained by the membrane method of curing. In general, the smoother or less absorbent the surface to which the compound is applied the more effective the curing will be.

VARIATIONS AMONG LABORATORIES

Although it is important that the results of laboratory tests be indicative of the service behavior of the material, it is equally important that the test methods be reproducible in different laboratories within a reasonable range of test error. It would be uneconomical to require that the manufacturer of curing compounds use different formulas to meet the requirements of each of the agencies purchasing it if it is to be used for the same purpose. This will be the case unless coordination and standardization of all details of the test methods are accomplished. It has been the aim in writing the method of test for water retention efficiency (C 156)[3] to avoid uncontrollable variables. To this end type I portland cement (some agencies specify type II) and graded Ottawa sand are required and the consistency of the mortar and water-cement ratio established as definitely as available laboratory apparatus will permit. Temperature and humidity of the laboratory used for making and testing specimens for the curing compounds are stated as are the temperature and relative humidity of the storage cabinet in which the specimens are placed during test. The exact moment at which the curing compound is to be applied to the surface of the mortar specimen is described as accurately as possible since this is of utmost importance in securing repeatability as well as reproducibility in and among laboratories.

During the past year Subcommittee III-g on Methods of Testing and Specifications for Materials Used in Curing Concrete of ASTM Committee C-9 on Concrete and Concrete Aggregates has conducted a series of cooperative tests among 15 laboratories using one commercial curing compound and a portland cement distributed with the compound. Each laboratory repeated the test using its own local brand of cement. Results of these tests have not been released by the subcommittee for publication since the tabulation has only recently been completed. Analysis of the data is still in progress but it can be stated that there were as many instances in which the distributed-cement mortar test specimens had the greater water loss through the membrane as there were local-cement specimens which had the greater loss. There was only one laboratory which found no difference. It is of interest that the range in results and the standard deviation for the distributed cement were greater than for the local cement. This would hardly be expected. It was anticipated, however, that the coefficient of variation would be high for the series and this was disappointingly true. For the distributed-cement test series, the average loss of water in grams per square inch was 0.1772 and the standard deviation was 0.1590 giving a coefficient of variation of 80.9. For the local-cement series the average loss was 0.1436 g per sq in. and the standard deviation 0.09167, making the coefficient of variation 63.7. It is obvious that the test for water retention efficiency is in need of improvement to assure better reproducibility.

STUDIES OF RELATIVE MERITS OF SEVERAL METHODS OF CURING

A positive means of evaluating the effectiveness of methods of curing has been the object of a considerable number of investigations and experimental projects both in the laboratory and the field (**6, 7, 8**). Many conflicting data have been recorded and with few exceptions satisfactory results have been reported with each method. The principal difficulties in coordinating laboratory and field results are the inability to control weather and construction conditions during the early stages of the field tests and in recording and interpreting the influences of climate and other deteriorating effects on the test sections during the intervals between condition surveys. Very little comprehensive research has been undertaken because of these difficulties and the expense of such studies. It should be mentioned, also, that when field studies of methods of curing are conducted the methods under comparison are each applied with the utmost care in order that no unfair advantage will be given. The average concrete job does not enjoy the same meticulous attention to curing and, to make a proper comparison of methods of curing, the average job performance should be the basis.

There are two general methods of curing: (1) one that keeps the concrete wet by the application of water and (2) one that keeps it damp by attempting to prevent the loss of the concrete mixing water. In the first category wet earth, wet burlap, wet cotton mats, and other media which must be sprinkled more or less frequently are employed. The second group uses paper blankets and the curing compounds. Each method is abused to varying degrees on the average jobs previously referred to—the first by failure to sprinkle often enough to keep water available for hydration of the cement and the second by not maintaining an impervious seal (rips and holes in paper blankets, missed spots with membrane curing, or early abrasion and scuffing of the membrane due to workmen and the public being unaware of the nature of the curing process). The methods in group 1 have the inherent advantage that they reduce the temperature changes in the concrete by both insulation and evaporation. Some paper blankets have sufficient reflectance to reduce temperatures in the concrete in hot weather and to provide some protection against freezing in moderately cold weather. The clear curing compounds offer no protection against temperature, and if a dye has been added to increase visibility the effect may be to increase heat absorption. This accounts for the increased use of pigmented curing compounds.

A recent survey of curing practices throughout the United States, conducted by the committee of the Highway Research Board on Curing Concrete Pavements, showed no decided preference for any one type of curing among the 53 agencies canvassed.

The first five methods in the list of 21 reported by the survey are shown here in tabular form with first, second, and third choice by the states or agencies expressing their preference:

Method	1st Choice	2nd Choice	3rd Choice
Cotton mats	7	6	3
Burlap followed by wet earth	6	2	..
Waterproof paper	5	5	3
Membrane	4	6	3
White pigmented membrane	2	1	2

The survey showed further that the membrane-forming compounds as a class exceeded all other methods in extent of use by a wide margin. Considering membrane types of curing only, of 42 agencies permitting its use 15 used only the clear type, 14 permitted only the pigmented, and 13 allowed either. It is doubtful that these preferences are based on more than personal opinion and meager observations in the majority of cases. Experience with pigmented compounds has been reported by Rhodes (**9**) and Peyton (**10**), the former reporting favorably and the latter unfavorably.

SUMMARY

Summarizing the efforts that have been made to evaluate curing compounds, it appears that most confidence is placed in the reasoning of the concrete technician and that this reasoning is supported by laboratory tests and field observations. There is need for much new and coordinated study of curing methods in the laboratory and field.

In the laboratory, standard methods for determining moisture loss through membranes, especially those formed by the curing compounds, are still to be developed even though the Tentative Method of Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156 – 52 T) is serving the purpose fairly well for many state and construction agencies.

Reflectance of the membranes and sheet materials can be measured by using the Standard Method of Test for Daylight 45-deg, 0-deg Luminous Directional Reflectance of Paint Finishes (D 771 – 47) or some simpler modifications (**11**). This test is coming to be used more extensively in order to minimize internal temperature variations.

No adequate method for detecting the deleterious effect which a liquid compound may have on the concrete has been generally adopted and there is a need for this.

Other properties of the compounds have no direct effect upon the efficiency of the curing although they are quite important to the user's economy, convenience, and comfort. Such properties include ease of removal, flammability, and toxicity.

In the field more extensive efforts should be made to secure data which will indicate the effect that the methods of curing have on structures and pavements—not by special projects involving elaborate organization and test procedure and including many varieties of curing methods, but by the use of two or three curing methods on many jobs followed by periodic observations and the averaging of the observations, thus minimizing the influence of construction and climatic conditions.

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DISCUSSION

Chairman A. T. Goldbeck.[1]—I was very much interested in one phase of this subject. We have had occasion in the past to coat a piece of plate glass with cutback asphalt. If that piece of glass is held up to the light, pinholes in the asphalt film are seen. When the coated glass is put in water, those pinholes begin to enlarge, and ultimately the film of asphalt will pull itself together and will form globules of asphalt on the surface. I am wondering if some of these compounds in drying will have that same tendency.

Mr. C. E. Proudley (*author*).—So far as I know, no one has ever looked at it that closely. My observations, which have been made by getting down on my knees and touching it with my fingers and looking at it as closely as I can, have not shown any tendency of that kind. Of course, on a concrete surface, you have the rough texture resulting from grains of sand, and this is comparable to mountain peaks and valleys when you are considering a thin film of compound of this kind. I imagine that the film was continuous very early in the life of the compound. The liquid compound, as it flows or settles before drying, will go to the bottom of the valleys between the peaks or points of protruding sand grains leaving numerous points of a vulnerably thin membrane which may be readily perforated as the compound ages.

One of the difficult items in the development of a test of curing compounds is the securing of a surface texture of the mortar specimens such as will always give comparisons on the same basis and with the same effective thickness of membrane.

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AN INVESTIGATION OF DESIGN METHODS FOR ASPHALTIC PAVING MIXTURES*

By B. A. Vallerga[1] and Ernest Zube[2]

For a number of years considerable discussion has centered on the question of whether any of the current methods for the design of asphaltic paving mixtures yield results that correlate reliably with performance. Many materials engineers hold the view that all the various design methods give a "right" answer in the hands of the "right" man most of the time and that in the final results there is little to choose between them. Whether or not the "right" answer is the result of chance or method is often regarded as a minor matter even though the use of more economical materials may have been excluded.

Among those individuals who consider this matter important, honest differences of opinion as to what constitutes a design method are prevalent. Consequently, there has evolved a large number of design methods ranging from very simple rules and formulas to very involved and comprehensive procedures. It is extremely difficult, if not impossible, to draw any conclusions regarding the relative merits of these many design methods because of the basic differences in philosophy from which each problem is approached. Moreover, even when design hypotheses are similar or even identical, procedures for preparing and processing test specimens may vary so widely as to give rise, in effect, to a number of "different" design methods.

Definition of Mix Design:

The following is submitted as an outline definition of the necessary, essential, and comprehensive steps to establish the design of an asphaltic paving mixture:

A. Select the kind of aggregate and the particle size distribution (grading).

B. Select the type and consistency (or grade) of asphalt.

C. Determine the relative proportions of asphalt and aggregate (*1*) by experience or empirical formulae, (*2*) by calculation using the void theory or the surface area theory, (*3*) by test.

It is to be implied that design criteria should be considered in each step and that the factors involved in making the selections are part of the basic philosophy of each design method. For example, one design method might consider the factor of aggregate shape (that is, crushed *versus* rounded) of extreme importance in selecting the type of aggregate in step A, whereas it might be disregarded entirely or considered of no consequence in another method.

Purpose of Investigation:

In this study a comprehensive correlation of various design methods was not intended; it was felt that a comparison of some of the more widely used design methods would be in order. Furthermore, for any one particular asphaltic paving mixture selected for the comparison pro-

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gram, the problem would be essentially that of comparing methods used to establish the design asphalt content.

Of the three approaches listed under *C* above, the first defies comparison because it involves the intangibles associated with judgment, as tempered by experience. Empirical formulas are generally based on grading analysis and represent the codification of experience. The second approach is fairly straightforward and involves two basically different points of view: one stating that the quantity of asphalt required in a paving mixture is a function of the void volume available between the particles of mineral aggregate and the other stating that the design asphalt content is a function of the surface area of the aggregate and the asphalt film thickness. Both theories are used extensively and one or the other is generally an integral part of most design procedures. The third approach involves the preparation of trial mixes with varying increments of asphalt content. Test specimens are fabricated and tested for stability and durability. On the results obtained, the mix exhibiting the best combination of the desired properties is selected and the corresponding asphalt content designated as the design value.

TABLE I.—SUMMARY OF DESIGN METHODS.

Design Method	Method of Establishing Design Asphalt Content		
	By Calculation	By Test	
		Method of Fabricating Test Specimens	Method of Testing for Stability
Hubbard-Field	Void theory	Manual tamping per standard procedure	Hubbard-Field extrusion device
Corps of Engineers	Void theory	Impact (100 blows, 10 lb at 18 in.)	Marshall apparatus
Bureau of Public Roads	Void theory	Double plunger (3000 psi)	Direct compression (ASTM D 1074 – 49 T)
State of California	Surface area theory	Kneading (150 tamps at 500 psi)	Hveem stabilometer and cohesiometer
Asphalt Institute	...	Double plunger (3000 psi) or kneading (250 tamps at 335 psi)	V. Smith triaxial cell

Most of the currently proposed design methods employ a combination of approaches (2) and (3) in the determination of a design asphalt content after the kind and grading of aggregate and type and grade of asphalt have been selected. Naturally, if it were not possible to obtain a paving mixture with these materials which would meet the established design criteria, there would be a change in grading of aggregate or asphalt viscosity, crushing, etc., or the mixture would be rejected entirely.

Table I lists five design methods currently in use and gives information regarding the methods used by each to select the design asphalt content. It should be noted that of the five methods, four use a combination of approaches (2) and (3).

It is the purpose of this investigation to compare these five methods with respect to:

1. The job asphalt contents which would be obtained on a given aggregate material of specified grading combined with a given type and grade of asphalt.
2. The methods used to evaluate the significant property of stability.[3]
3. The effect of changing compaction methods on the results obtained by the various mechanical stability tests.

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[3] The term stability as used here means, specifically, the resistance to deformation of an asphaltic pavement under traffic loads.

TABLE II.—TRAFFIC VOLUMES, U. S. HIGHWAY 101, SOLEDAD TO GONZALES.

Year	Total Vehicles	Daily Traffic Volume			
		Passenger Cars		Trucks, Trailers, and Busses	
		Number	Total, per cent	Number	Total, per cent
1935[a]............	3 926	3 139	80	787	20
1951[b]............	6 946	5 787	83	1 159	17

[a] Traffic count for one 24-hr period, Monday, July 15, 1935.

[b] Average daily traffic, Report of California Division of Highways, 1951.

TABLE III.—SUMMARY OF AGGREGATE GRADINGS.

Sieve[a]	Specifications (1935 Standards)	Preliminary Design (1936)	Control[b] after Extraction (1936)	Experimental (1952)
1 in....................	100	100	100	100
¾ in....................		88	91	90
½ in....................		70	...	77
No. 3..................	52–66	65	63	66
No. 10................	36–48	45	47	47
No. 20................		31	...	32
No. 30................		26	...	26
No. 40................	20–30	23	25	22
No. 80................		18	18	15
No. 200..............	6–11	10	8	9
No. 270 (wash).........		8	...	8

[a] Sieves are those used during construction in 1936.

[b] Average of eleven control samples (see Table V).

TABLE IV.—TEST RESULTS ON ROMC-5 LIQUID GRADE ASPHALT.

Test Conducted	Specification Limits (1935 Standards)	Range of Test Values[a] (1936)	Experimental Asphalt (1952)
Flash Point, deg Fahr min.....	150	150+	150+
Viscosity			
Saybolt Furol at 180 F	170–280	175–280	196
Distillation, per cent by volume			
Total to 437 F, max per cent	1	0	2
Total to 600 F, max per cent	11	0.05–5.5	8
Total to 680 F, max per cent	15	4–14	10
Test of residue from distillation			
float test at 122 F..........	100–250	134–242	233
Approximate amount of kerosine			
per cent....................	11	10	10

[a] Range indicated is for a total of 25 samples.

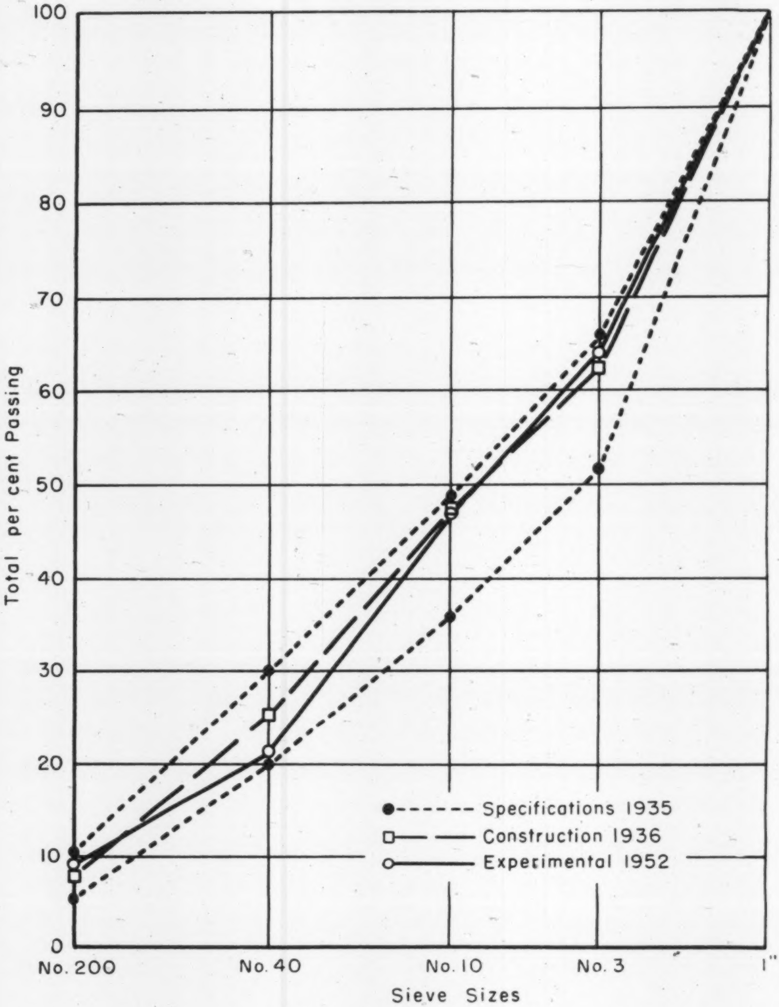

FIG. 1.—Grading Curves.

TABLE V.— PRELIMINARY DESIGN DATA FOR PLANT-MIXED SURFACING.
Test No. 9864, August 13, 1936.

Grading Analysis as Used	
Sieve, in.	Per cent Passing by Weight
1 in.	100
No. 3	65
No. 10	45
No. 40	23
No. 200	10

Note.—Grading represents blend of
37 per cent crushed granite (1 in. × No. 3)
55 per cent distintegrated granite (passing No. 3)
8 per cent disintegrated granite (passing No. 40)

Surface area, 45 sq ft per lb

Asphalt content—weight aggregate, per cent	3.0	4.4	4.7	5.0
Stabilometer values[a]	37	38	39	34
Bulk specific gravity[b]	2.35	2.37	2.38	2.38
Swell test, in.		0.002		
Permeability, ml per 24 hr		50		

[a] Stabilometer value of 35 min specified in California for primary highways.
[b] Specimens fabricated by a combination of impact and 3000 psi static pressure.

TABLE VI.—CONTROL TESTS ON FIELD SAMPLES OF PLANT-MIXED SURFACING.
September, 1936.

Test Number	Date Sampled	Asphalt Content, per cent by extraction	Per Cent Passing							Specific Gravity	Stabilometer Value, per cent	Swell, in.	Permeability, ml per 24 hr	Water Content, per cent
			1-in. mesh	¾-in. mesh	No. 3	No. 10	No. 40	No. 80	No. 200					
10630	17	4.0	100	91.8	58.1	43.4	23.9	18.2	8.9	2.38	38	0.003	20	0.8
10631	18	4.3	98.6	93.2	58.3	44.4	23.8	17.5	7.9	2.38	53	0.010	30	0.7
10694	21	4.3	100	93.0	62.5	48.9	25.2	18.1	7.9	...	36	0.005	50	0.7
10693	22	4.4	100	90.8	66.7	52.3	24.3	17.5	7.4	2.36	32	0.000	35	0.6
10712	23	4.6	100	95.1	70.6	55.0	27.8	19.5	7.4	2.35	40	0.003	35	0.8
10743	24	4.2	100	87.9	62.7	45.0	22.2	17.6	7.6	2.36	40	0.001	50	0.6
10744	25	4.1	100	92.8	66.4	49.0	24.9	17.5	7.5	2.41	37	0.005	25	0.8
10754	26	4.4	100	88.6	59.5	43.7	22.0	16.7	6.5	2.35	45	0.001	5	0.6
10755	28	4.1	100	83.3	54.6	42.0	23.5	17.8	8.1	2.35	51	0.001	5	0.8
10872	29	4.1	100	88.0	60.9	47.0	24.0	17.4	7.8	2.34	52	0.003	10	0.7
10915	30	4.2	100	90.6	66.7	52.2	35.2	18.2	8.2	2.40	47	0.000	40	0.9
Average		4.2	99.9	90.5	62.5	47.5	25.2	17.8	7.7	2.37	43	0.003	25	0.7

TABLE VII.—TYPICAL DAILY PLANT REPORTS.
Plant-Mixed Surfacing.

	August 15, 1936	October 1, 1936
Grading analysis,[a] per cent passing by weight		
Sieve		
1 in.	100	100
No. 3	60	59
No. 10	45	47
No. 20	32	36
No. 40	22	27
No. 80	16	15
No. 200	7.8	10.6
Asphalt content,[b] per cent	4.2	4.4
Temperatures, deg Fahr		
Asphalt	220	205
Aggregate	185	210
Mixture	185	200

[a] 60 per cent sand and 40 per cent crushed granite (Sand is disintegrated granite from borrow pit 500 ft west of Station 1 + 60).
[b] By dry weight of aggregate (ROMC-5 asphalt).

The material selected for this comparison program is a densely graded disintegrated granite combined with a road oil medium cured liquid asphalt (ROMC-5). Selection of this material was predicated on the following:

1. A section of main highway in California had been constructed during the summer months of 1936 with this plant-mixed surfacing material and as of 1953, after seventeen years of continuous service under main-line traffic, the pavement is still in excellent condition.
2. Original design recommendations and both plant and laboratory control data are a matter of record.
3. Locations of the original sources of materials were well established and were readily accessible.
4. The low viscosity characteristics of the ROMC-5 asphalt, even after extended curing, would tend to magnify differences between stability test methods.
5. Field specimens were readily obtainable by field coring operations.

Description of Prototype:

The asphaltic pavement constructed of the material used in this study is located in California Highway District V in Monterey County between the towns of Soledad and Gonzales and is part of the existing coast highway, U. S. 101. Constructed during the months of August to October in 1936 under contract 85 - TC3, the project covered a total distance of 8.3 miles and cost about $140,000. At that time the operation consisted of blanketing an old, badly cracked concrete pavement with 2½ in. of plant-mixed surfacing and constructing an entirely new roadway on line and grade changes. In the latter case, crusher-run base was imported and covered with 2½ in. of the same plant-mixed surfacing. Because of the narrow width of the existing concrete pavement (15 ft) compared to the new highway width of 36 ft (including shoulders), additional base preparations were required along the road adjacent to the old concrete pavement.

Table II is a summary of the actual traffic-count values in 1935 and in 1951.

Construction procedures called for the application of a prime coat of slow curing liquid asphalt (SC-2) to the surface of the crusher-run base and medium curing liquid asphalt (MC-2) to the existing concrete before placing the surfacing material. The asphaltic paving mixture was manufactured by plant mixing a blend of approximately 60 per cent by weight of fine disintegrated granite with 40 per cent by weight of coarse crushed granite rock and adding between 4.2 to 4.4 per cent ROMC-5 liquid grade asphalt by weight of dry aggregate. The fine material was obtained from a local borrow pit located 500 ft west of Station 1 + 60, Section C, and the crushed granite imported from the Logan quarry near Watsonville. In Table III are the gradings of the combined aggregate material at various stages of the operation together with the grading selected for use in the test program of this investigation. Figure 1 illustrates the characteristic shapes of the grading curves and the similarity between the grading used in construction and that used in this study.

Test results on the asphalt used in construction are presented in Table IV which also includes the 1935 California specification for ROMC-5 liquid asphalt. Current specifications no longer include this type of asphalt and, therefore, a special blend of SC-6 (approximately 350 penetration) and 10 per cent kerosine was prepared for the experimental series of tests. Test values for this experimental asphalt are also included in Table IV. From an examination of the tabulated values, a very close agreement in test values between the experimental asphalt and the asphalt used in construction is evident.

Prior to construction a preliminary mix design was made by the Materials Laboratory of the California Division of Highways on samples of the aggregate and asphalt. Normal test procedures (**1**)[4] involved the testing of laboratory specimen in the Hveem stabilometer to evaluate the stability and to establish the design asphalt content. Table V indicates the tests that were performed and contains the essential test results obtained. From an analysis of the data by the California procedure, which involves computing the product of surface area and asphalt film thickness, the design asphalt content appeared to be about 4.4 per cent. It was therefore recommended on the preliminary report that the asphalt content should be between 4.4 and 4.7 per cent by weight of dry aggregate. For this range of asphalt content stabilometer values exceed the minimum of 35 required for asphaltic pavements subjected to main-line traffic in California.

TABLE VIII.—OUTLINE OF TEST PROGRAM.

Design Method	Compaction Methods	Stability Test Method
State of California	Kneading (150 tamps at 500 psi)[a] Double plunger (2000 psi) Impact (100 blows, 10 lb at 18 in.)	Stabilometer and cohesiometer
Hubbard-Field	Hubbard-Field[a] Kneading (150 tamps at 350 psi)	Hubbard-Field (extrusion)
Bureau of Public Roads	Double plunger (3000 psi)[a] Kneading (180 tamps at 500 psi)	Direct compression (ASTM D 1074 – 49 T)
Corps of Engineers	Impact (100 blows, 10 lb at 18 in.)[a] Kneading (150 tamps at 500 psi)	Marshall apparatus
Asphalt Institute (triaxial)	Double plunger (3000 psi) Kneading (250 tamps at 335 psi)[a]	V. Smith triaxial cell

[a] These compaction methods are standard for the corresponding stability tests.

During construction, field control samples were taken periodically and checked for compliance with specifications. Eleven such samples are shown in Table VI with a summary of the test results obtained. In general, the results indicate high stability and good durability characteristics as evaluated by the stabilometer and swell tests. Careful controls were also exercised over the plant mixing operations. Data from two typical daily plant reports are given in Table VII. Both Tables VI and VII serve to illustrate the degree of uniformity obtained in producing the plant-mixed surfacing and the low mixing temperatures used.

Experimental Program:

With the above backlog of data and information available, a program of testing was initiated to obtain further data on which comparisons of the five design methods could be made. Sufficient quantities of materials were obtained to supply the needs of a testing program which included the molding of at least two specimens at five asphalt contents by two or more compaction methods for test by each of the five methods given in Table I. An outline of the testing program is given in Table VIII, indicating the methods of specimen preparation used and the tests conducted. Increments of 1 per cent asphalt content were used and ranged from 3 to 7 per cent by weight of aggregate, except for the Asphalt Institute triaxial method where the range was from 4 to 8 per cent, inclusive.

In addition to the above laboratory program, a number of field cores were taken from the section of highway be-

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1095.

TABLE IX.—SUMMARY OF TEST RESULTS ON FIELD CORES.

	V Monterey - 2C						V Monterey - 2D				
Core number	CF - 1	CF - 2 CF - 3 CF - 5 CF - 6	CF - 4	CH - 1	CH - 2	CH - 6 CH - 7 CF - 8	CG - 1	CG - 2	CG - 3	CG - 5	CG - 6
Location by station number	73 + 82	73 + 99 74 + 09 80 + 15 80 + 26	80 + 04	170 + 35	170 + 40	240 + 36 240 + 45 240 + 57	566 + 90	567 + 04	567 + 14	573 + 12	573 + 22
Base	CRB	CRB	CRB	Concrete	Concrete	Concrete	CRB	CRB	CRB	CRB	CRB
Field density (bulk sp gr)	2.41	2.42 (Total)	2.35	2.43	2.45	2.50 2.46 2.48	2.41	2.45	2.41	2.43	2.40
Asphalt content, per cent (by extraction)	4.2	...	4.6	...	...	... 4.1 ...	3.9	...	4.2	...	...
Air voids, per cent	4.8	...	6.4	...	...	3.7	5.1	...	4.8	...	...
Stabilometer value	37	...	36	23	42	...	31	37	31	...	...
Cohesiometer value	317	...	260	493	620	...	265	290	317	...	...
Marshall test Stability, lb Flow, 1/100 in.										 1505 15½	 1290 14½
V. Smith triaxial Angle of friction—0 Cohesion—C, psi		 49 deg 6									 ... ...

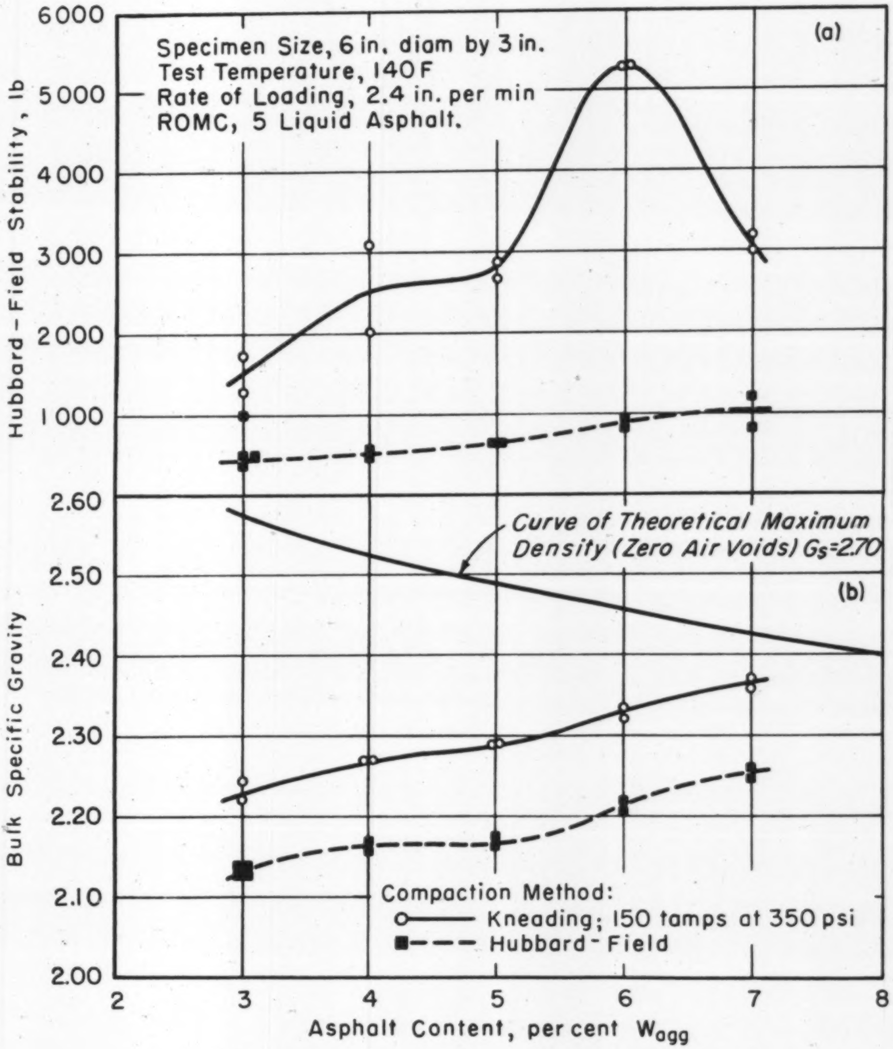

FIG. 2.—Hubbard-Field Test Results, Soledad-Gonzales Aggregate.

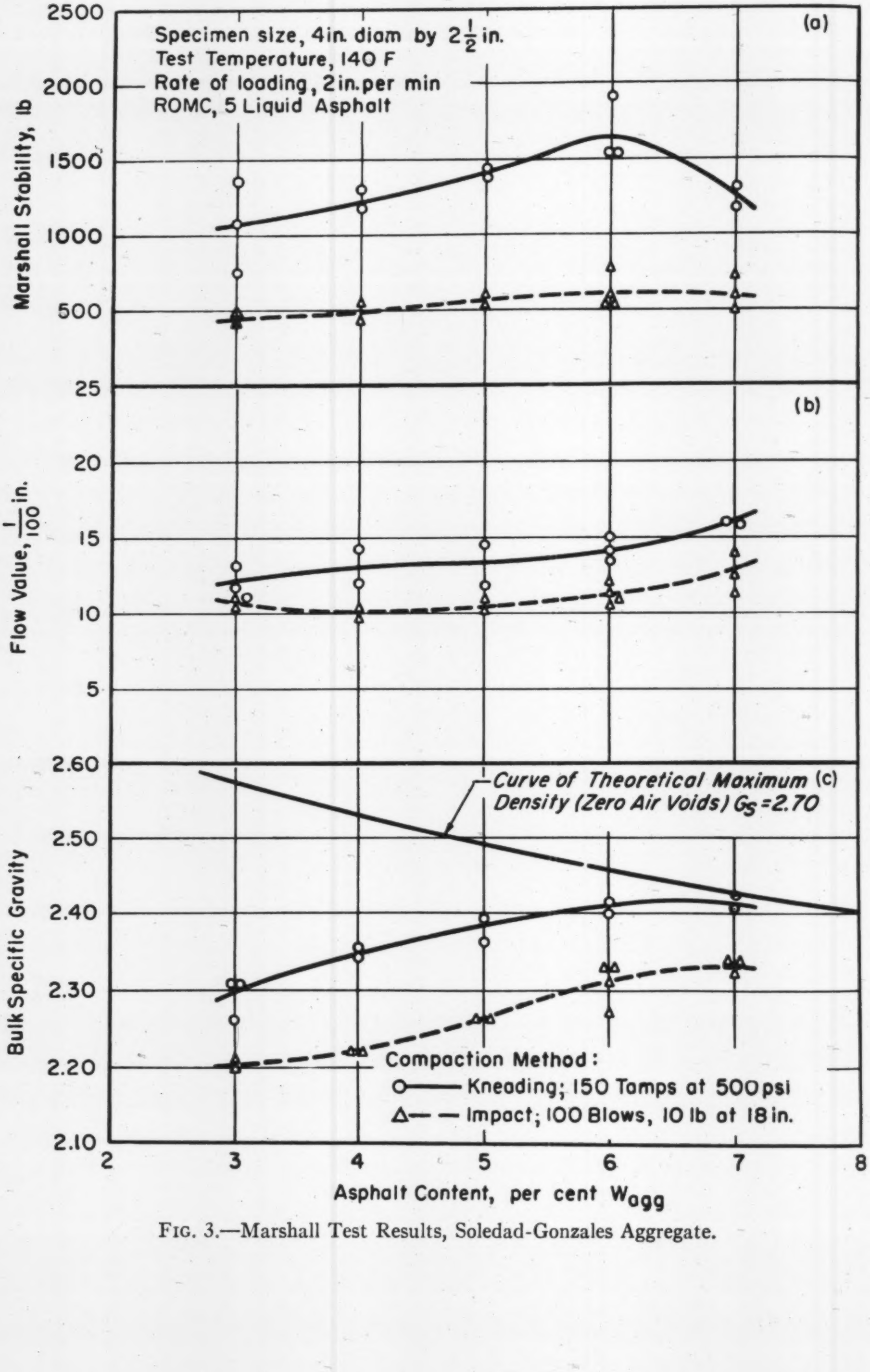

FIG. 3.—Marshall Test Results, Soledad-Gonzales Aggregate.

tween Soledad and Gonzales. These cores were cut with diamond bits 4 in. and 5 in. in diameter. Table IX is a listing of the cores taken and their locations. After cutting with a diamond saw, the cores were subjected to bulk specific gravity determinations and other tests, depending on the suitability of the cores for such purposes. Because of the variation in core size from the standard stabilometer specimen, several cores were coated with a thin layer of plaster of Paris before testing. Also included in Table IX is a summary of all results obtained on all tests conducted on any of the core specimens.

As the work progressed, it became evident from an inspection of the accumulating data that the field core densities were considerably higher than any obtained in the laboratory by any compaction method at the corresponding asphalt content. Therefore, in order to obtain an indication of what laboratory compactive effort was required to duplicate field densities in this case, it was decided to prepare four identical mixtures of the Soledad-Gonzales aggregate at an asphalt content of 4.5 per cent and to subject each mix to kneading compaction with an increasing number of blows on a logarithmic scale (that is, 100 1000, 10,000, and 50,000 tamps) while maintaining the tamper foot pressure at 500 psi.

Preparation of Test Specimens:

Identical procedures were employed in the preparation of the asphaltic mixture, regardless of the compaction method to follow. Blending, proportioning, mixing, and curing operations were carefully controlled and the temperature during mixing, in all cases, was maintained between 200 and 230 F. Mixing was performed in a mechanically driven vertical bowl mixer (45 rpm) for a period of five minutes, after which the mixture was spread in a shallow pan and allowed to cure in a forced draft oven for a period of 15 hr at a temperature of 140 F.

After the curing operation the predetermined quantities of mixed material to give specimens of desired proportions as required in each design procedure were measured and the test specimens were molded. The compaction procedures commonly associated with given test procedures were followed very closely. These procedures are described in the literature for each design method (**3, 4, 5, 7**) and in ASTM Method D1074.[5] All specimens were molded at a temperature between 200 and 230 F.

Following compaction, the molded test specimens were allowed sufficient time to meet the test temperature requirements for each test method. Procedures for testing were also those intimately associated with the given design method. Information with regard to other vital statistics, such as test temperatures, rates of loading, size of specimen, etc., is included as part of the figures illustrating the test results, which follow.

Analysis and Discussion of Test Results:

Data from the Hubbard-Field test plotted in Fig. 2 indicate that kneading compaction gives a higher density than standard Hubbard-Field compaction. A pronounced increase in Hubbard-Field stability is apparent with kneading compaction. With Hubbard-Field compaction the Hubbard-Field test rates this mix very poorly in terms of stability. The optimum asphalt content based on the Hubbard-Field criteria, assuming that the material would not be rejected for low stability, would be in excess of 7.0 per cent. With kneading compaction the design asphalt content by the same criteria would be reduced to about 6.0 per cent.

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[5] Tentative Method of Test for Compressive Strength of Bituminous Mixtures (D 1074 - 52 T), 1952 Book of ASTM Standards, Part 3, p. 1270.

In Fig. 3 the regular Marshall test results would be taken to indicate that this is a material of questionable stability because the maximum Marshall stability under the standard method of compaction was only about 700 lb. Kneading compaction improves the stability as evaluated by this test method. Based on the Corps of Engineer criteria for selection of design asphalt content, it is calculated that the design value at 5 per cent voids would be around 6.3 per cent, assuming the mix to be stable.

Another similar pattern is evident in Fig. 4, where the data for the Bureau of Public Roads design method are presented. Kneading compaction again changes the position of the stability curves in Fig. 4 (*a*) quite markedly. Also, density by kneading is higher than that obtained by 3000-psi double-plunger pressure. According to these data the asphalt content by the Bureau of Public Roads design method based upon 4 per cent air voids would be around 6.0 per cent.

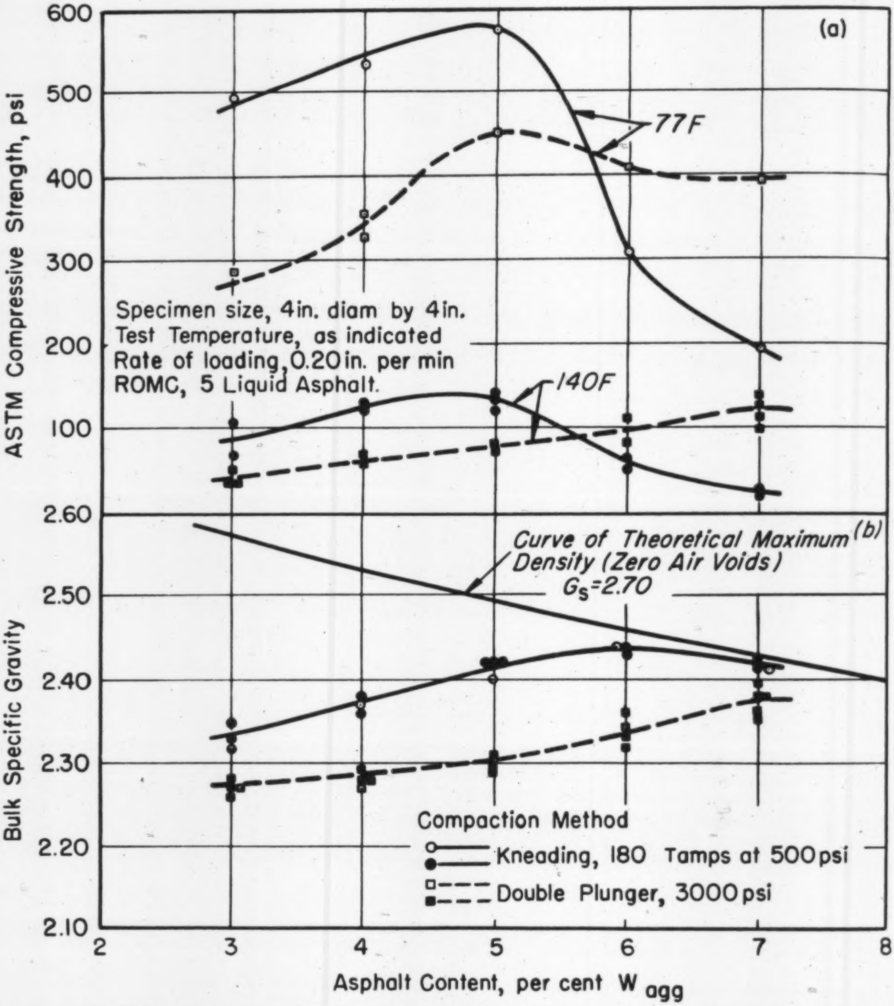

FIG. 4.—ASTM Compression Test Results, Soledad-Gonzales Aggregate (Bureau of Public Roads Design).

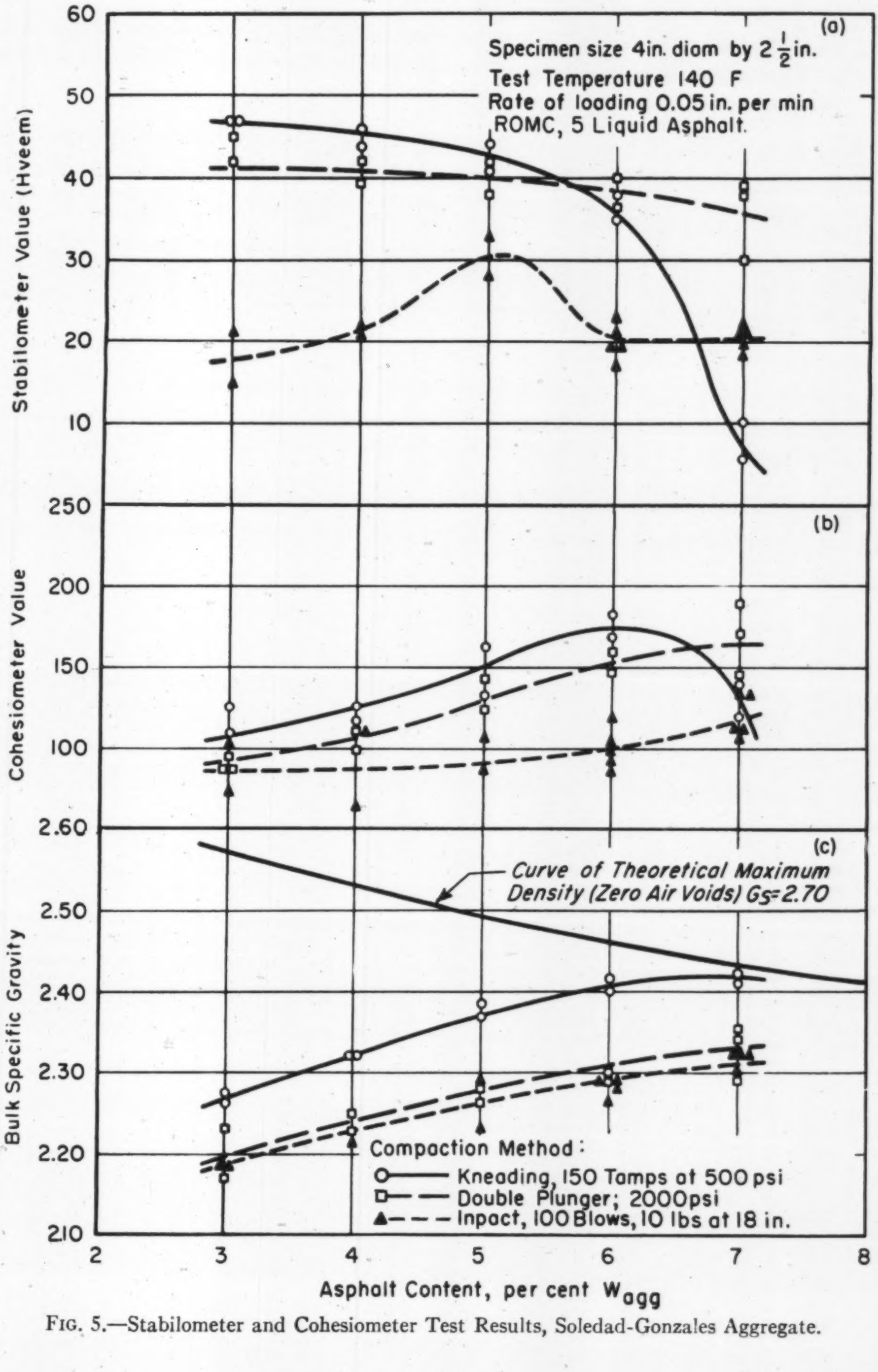

FIG. 5.—Stabilometer and Cohesiometer Test Results, Soledad-Gonzales Aggregate.

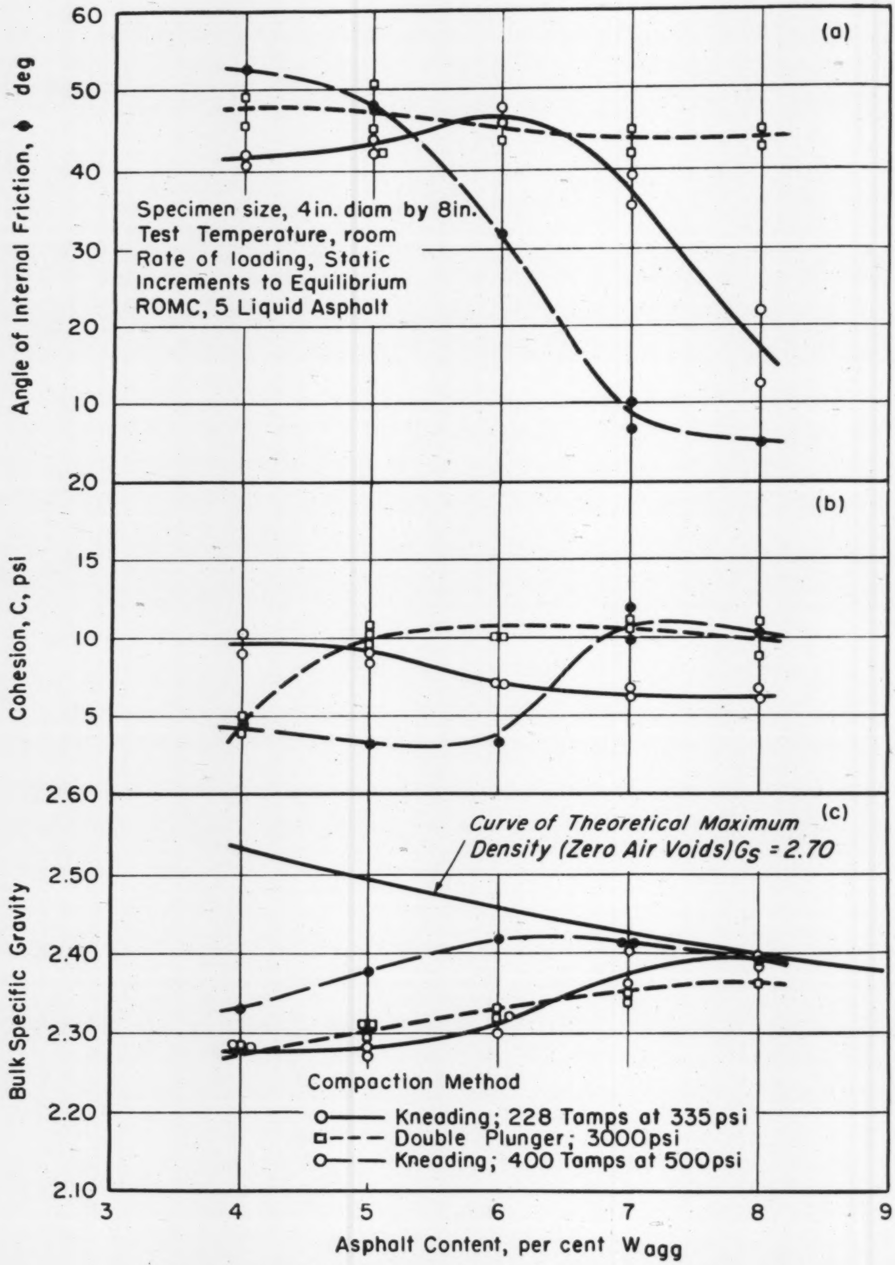

FIG. 6.—Smith Triaxial Test Results, Soledad-Gonzales Aggregate.

Figure 5 presents the results obtained in the California method of test with three compaction methods. By both double plunger and kneading compaction, the asphaltic paving mixture shows high stability as evaluated by the stabilometer. Cohesiometer values are rather low, as one would normally expect from a mixture made with a low viscosity asphalt. Figure 5 (*c*) clearly indicates that kneading compaction is more effective in obtaining high densities than are the other two compaction methods. At the present time, with specimens made with the kneading compactor, it is probable that a somewhat higher asphalt content would be recommended as the stability values are adequate up to 5 per cent asphalt content. By double plunger compaction, stability values are good up to 7 per cent, which is not reasonable.

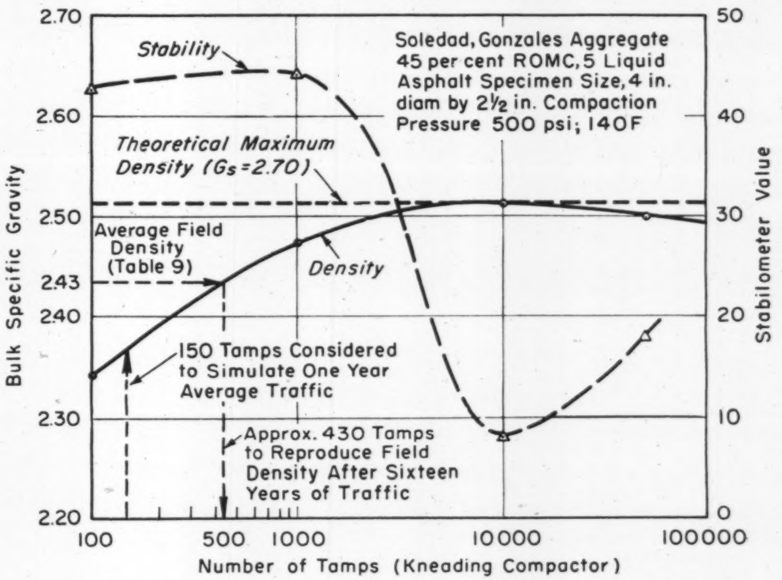

FIG. 7.—Effect of Prolonged Kneading Compaction on Density and Stability.

The fifth and last design method considered in this investigation, the Asphalt Institute triaxial (Fig. 6), shows a relationship between kneading and double plunger compaction similar to that obtained in the Hveem stabilometer. However, the lower compactive effort in the kneading process recommended for these specimens results in a density considerably lower than that obtained by kneading compaction on all other size specimens. It should be noted, further, that the range of asphalt contents had to be increased by 1 per cent in order to obtain a well-defined stability curve. A greater compactive effort by kneading, as shown in Fig. 6, did increase the density values and cause a change in position of the stability curve. Using kneading compaction normally used a design asphalt content of 6 per cent is indicated, whereas with increased kneading compaction the design asphalt content decreased to about 5.0 per cent. Double plunger compaction mixes up to 8 per cent show good stability.

A comparison of the data presented in Figs. 2 to 6 with the limited field core data in Table IX reveals several significant points. Field densities from this pavement under traffic for 17 years, for example, are invariably higher than laboratory densities on freshly prepared mixtures. It is evident from an inspection of Fig. 7, which contains the results of a series of specimens subjected to repeated tamping in the kneading compactor that increasing the number of load applications increases the density. The operation of the kneading compactor was originally adjusted to simulate about one year's traffic on the average road. (150 tamps at 500 psi on a 4-in. diameter by $2\frac{1}{2}$-in. specimen was established in 1937.) Notice, also, the effect of additional compaction on the stabilometer value which, for this particular mixture with 4.5 per cent asphalt, gradually increased and then dropped markedly with further compaction.

TABLE X.—COMPARISON OF TEST AND DESIGN METHODS BY COMPARING ASPHALT CONTENTS.

Design Method	Asphalt Contents, per cent			
	For Maximum Stability		For Design	
	Compaction normally used	Kneading	Compaction normally used	Kneading
Hubbard-Field	over 7.0	6.0	over 7.0	about 6.0
Corps of Engineers	6.2	6.0	about 6.3	about 5.5
Bureau of Public Roads	5.2	4.9	about 6.0	about 4.7
State of California	less than 3.0	less than 3.0	about 5.0	about 5.0
Asphalt Institute triaxial	about 4.0	less than 4.0	about 6.0	about 5.0

Secondly, the stabilometer values of the cores compare quite favorably with the present-day test results considering the different factors involved. Moreover, in both cases the test results indicate that the pavement should be capable of supporting the traffic loads.

Thirdly, the cohesiometer values of the compacted mixture in the field increases with time on the road. This is due probably to a combination of increasing density due to the compacting action of traffic and the hardening or aging of the asphalt over a 17-yr period. A specimen of asphalt recovered by the Abson procedure from cores CH - 6, CH - 7, and CH - 8, however, gave a penetration value of 231, which indicates that the amount of age hardening in this pavement was not excessive. The penetration of the base asphalt of an ROMC-5 is generally about 350 before the kerosine is added.

Marshall test results in cores CG - 5 and CG - 6 compare quite favorably with the laboratory results on specimens prepared by kneading compaction, which would seem to indicate that kneading is more representative of field compaction, especially since the asphalt in the field still has a fairly high penetration value (231). Similarly the V. Smith triaxial cell obtains good correlation between laboratory prepared specimens and a composite field specimen made by stacking cores CF - 2, CF - 3, CF - 5 and CF - 6.

Finally, the air void contents of the several core specimens definitely indicated that the mix in the field is not in danger of approaching saturation, and no bleeding or loss in stability should be expected. A number of other tests have been run with other core specimens; the data are included in Table IX.

Summary and Conclusions:

A dense-graded plant-mixed surfacing made with a liquid grade asphalt was used to construct an asphaltic pavement which has been in continuous service for

17 years on a main highway in California. Although subjected to heavy traffic, this pavement has performed excellently with a normal amount of maintenance and is still in good condition.

Because of its excellent performance record and the low viscosity characteristics of the asphalt, a similar paving mixture was selected for a comparison program of pavement design methods and stability test methods. On the basis of the test results obtained on both laboratory and field specimens the following conclusions are drawn:

1. An asphalt content of between 4.2 and 4.7 per cent was actually used in construction and there is no evidence to indicate that any more (or less) asphalt should have been included, at least not more than 5 per cent by weight of dry aggregate.

2. The reliability of any stability test method that indicates a maximum stability value at an asphalt content in excess of 5 per cent should be questioned, therefore, either as to (*a*) the adequacy of the test specimen in representing a field compacted specimen, or (*b*) the validity of the test method in evaluating the property of stability (or resistance to deformation under load). From Table X it is evident that the Marshall, Hubbard-Field and direct compression tests and even the Smith triaxial cell are in this questionable category when the methods of compaction commonly associated with these tests methods are used. In general, kneading compaction decreases the asphalt content at maximum stability, but only in the cases of the direct compression test and the Smith triaxial test was the decrease sufficient to come within the 5 per cent maximum established. It is evident from Fig. 6 that the Smith triaxial asphalt content at maximum stability also decreased to less than 5 per cent when the 4-in. diameter by 8-in. specimen was thoroughly compacted by kneading action in order to approach more closely the field densities.

3. A comparison of probable design asphalt contents based on criteria generally employed is also included in Table X and indicates that most commonly used design methods recommended the use of too much asphalt for the kind and grading of aggregate and type of asphalt used in this investigation.

Acknowledgment:

To all those who assisted in obtaining the data, the writers wish to express their gratitude, especially Messrs. Dale Warren, Raymond Lundgren, Peter Hsu, and Robert Cooper.

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DISCUSSION

Mr. C. A. Carpenter.[1]—The paper by Messrs. Vallerga and Zube is a valuable contribution in the development of the science of designing and testing bituminous paving mixtures. It utilizes the realistic approach of selecting an example of pavement having a long and satisfactory service record and of correlating all the available data on the mixture in the road with test data on a similar mixture prepared in the laboratory. A correlation of this sort is most convincing and useful, particularly when, as is the case here, the data give every indication of careful work in the laboratory.

It is felt that more emphasis might well be placed on certain of the indications that are quite apparent from the data presented:

1. The stabilometer values for the range of trial asphalt contents used in arriving at the original design, Table V, reached a maximum at 4.7 parts of asphalt for 100 parts of aggregate by weight and, when plotted, indicate that nearly maximum stability could be expected for asphalt contents of about 4 to just under 5 parts. Table VI shows that this is the range that was actually used in the road which showed good serviceability for 17 yr. The fact that the road is still in excellent condition after 17 yr of service does not, of course, prove that these were the only asphalt contents that would have been satisfactory, but it is adequate proof that these specific asphalt contents were satisfactory and, therefore, that the maximum stabilometer values (1936 method) could safely be considered to indicate the optimum asphalt content. Thus, the stabilometer, as used in 1936 with the combined tamping and compression process for molding the specimens, see note Table V, apparently predicted the correct asphalt content very accurately while also reflecting the good quality of the mixture.

2. It appears from Fig. 4(*a*) (upper solid line and upper dash line) that the ASTM Tentative Method of Test for Compressive Strength of Bituminous Mixtures (D 1074 – 52 T)[2] would give an equally dependable evaluation of the correct asphalt content regardless of whether the specimens were compacted by direct compression or kneading. The specimens compacted by direct compression show compressive strength values at 77 F in the high range 400 psi plus, which are believed to be indicative of adequate stability for heavy truck traffic. The optimum asphalt content is indicated to be 5 parts ± 0.2 per 100 parts of aggregate, which is a very good check with the results obtained orginally (1936, Table V) with the stabilometer and in the current study by stabilometer using straight impact molding (Fig. 5(*a*) short-dash line). Thus it appears that combined tamping and compression (the design method), spading and compression, ASTM method, kneading compaction and impact tamping are all four capable of producing specimens that are sensitive to bitumen content as indicated by

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[1] Senior Highway Research Engineer, Physical Research Branch, Bureau of Public Roads, Washington, D. C.

[2] 1952 Book of ASTM Standards, Part 3, p. 1270.

stability, although the degree of kneading compaction in present use by the authors appears to render the stabilometer ineffective for determining asphalt content.

3. It is quite evident that the kneading compactor used by the authors consistently produces a greater degree of compaction in this type of mixture than any of the other methods employed in the study. This is true even for the mixtures containing a considerable excess of asphalt. This would appear to indicate that greater compaction could be expected on other types of mixtures by the kneading compaction procedure than is obtained by the other methods. This constitutes a strong argument in favor of the kneading compactor since it permits closer approach to, if not complete attainment of, the high densities obtainable in testing the unbituminized aggregates by vibration and is probably capable of reproducing almost any degree of field compaction. The laboratory of the Bureau of Public Roads now has a kneading compactor built from plans furnished by Mr. Vallerga and is carrying out a correlation study of test results on cores from pavements currently being built *versus* test results on laboratory molded specimens of the fresh paving mixtures using standard ASTM molding procedure, various degrees of kneading compaction, and other procedures. At present this study is being made only on typical hot-mix construction in the vicinity of Washington, but in the light of the very interesting data on a liquid asphaltic mixture presented in the paper under discussion, it might be concluded that laboratory compaction and testing procedure satisfactory for surfacing containing penetration grade asphalt may not be satisfactory for the mixtures containing very soft asphalt or cutbacks.

An important relation that generally exists in bituminous mixtures, that between stability and the density of the aggregate portion of the compacted mixture, is so often overlooked that some restudy of the authors' data on this relation has been made.

In Fig. 4 the density curves for kneading compaction and for double plunger compression are plotted on the basis of over-all compacted mixture density *versus* asphalt content. This is fairly common practice, but curves so plotted have never been known to correlate in any way with stability. They show only the over-all changes in bulk specific gravity which provide no direct measure of the very important underlying factor affecting stability, the degree of compaction of the aggregate particles within the mixture.

The statement that density *per se* does not affect stability has been made repeatedly. This is true as long as the word "density" is confined in its meaning to the classic definition, mass divided by volume, where the "mass" includes all the materials in the mixture. In order to correlate density with stability, a regular procedure in the laboratory of the Bureau of Public Roads, it is necessary to consider the density of the aggregate portion of the mixture. This is easily determined by computation when the overall or "classic" density has been determined and the ratio of asphalt to aggregate is known. The following examples illustrate the ease of the computation:

Example A

Mixture contains 6 parts of asphalt by weight to 100 parts of aggregate.

Density (bulk specific gravity) of compacted mixture 2.420 g per cu cm.

Density of aggregate portion 2.420 $\times \frac{100}{106}$ = 2.283 per cu cm.

Example B (same mixture as A)

Mixture contains 5.66 per cent asphalt. (5.66 parts by weight to 100 parts of total mixture).

Density (bulk specific gravity) of compacted mixture 2.420 g per cu cm.

Density of aggregate portion $2.420 \times \frac{94.34}{100} = 2.283$ g per cu cm.

All of the densities plotted in Fig. 4 were converted as in Example A to aggregate densities and replotted to determine where, on the basis of aggregate density, the maximum stability might be expected to occur. In the series of specimens compacted by kneading, the maximum aggregate density occurred at 5 parts by weight of asphalt to 100 parts by weight of aggreate. The computed aggregate densities were as follows:

Asphalt Content Parts	Aggregate Density, g per cu cm
3	2.27
4	2.29
5	2.30
6	2.29
7	2.25

In Fig. 4 the compression test values for kneading compacted specimens also reached a maximum at 5 parts asphalt, or slightly less, perhaps 4.8, as judged by the curve. Thus, the authors' data show the usual good correlation between maximum aggregate density and maximum stability. The very rapid fall of the stability curve after the aggregate density starts to fall is also characteristic and indicates the passage from an interlocking coarse aggregate system with the bitumen only filling interstices between the aggregate particles to a mastic system with the coarse aggregate particles partially separated from each other by the mastic and with fewer and fewer points of contact as the proportion of mastic and with fewer and fewer points of contact as the proportion of mastic is increased by increasing the asphalt content.

These trends show clearly in compression tests with specimens compacted by spading and direct compression for aggregates containing natural or processed artificial sand as the intermediate aggregate, and the densities and the stability criteria correlate well with the field on hot-plant mixtures as generally laid and compacted in the field. They do not show as sharply and, in some cases not at all, when the entire aggregate grading is made up from crushed materials, including unprocessed stone sand. This is illustrated when the aggregate densities are computed from the lower curve in the lower part of Fig. 4 representing mixture densities as obtained by compression molding. When the aggregate densities so obtained are plotted, the result is a straight horizontal line for all asphalt contents from 3 to 6 parts and an insignificant rise at 7 parts. Thus, since there is no appreciable difference in aggregate density, the rise of the stability curve (dash line, upper section of Fig. 4) up to optimum merely indicates the increasing cohesive effect of the greater quantities of asphalt. Since the hot-mix surfacings do not show the great increases in density under traffic indicated for the liquid asphalt mixtures by the Soledad-Gonzales surfacing mixture, the attitude has been taken that this type of mixture made up entirely of crushed aggregate particles is generally so stable by nature that the asphalt content is not a critical factor within rather wide limits in hot-plant mixtures. However, it appears from California's experience with the Soledad-Gonzales road where liquid asphalt was used that it would be unsafe to make this assumption for liquid-asphalt mixtures. There is some indication that it is no longer a safe assumption for hot-plant mixtures because of the rapid increase in truck traffic on many routes.

The kneading compactor, or perhaps the combined tamping and compression molding procedure used in the design for the Soledad-Gonzales road surfacing, would apparently give a more conservative design basis for all bituminous mixtures than does the present spading and compression procedure. The more conservative design seems to be well justified for the type of mixture used on the Soledad-Gonzales road. An effort should be made to obtain a better understanding of the importance of higher initial compaction in the construction of hot mixtures than is generally obtained under present construction procedure. Recent experience with hot-plant mix surfacing on arterial routes that are subjected to high percentages of truck traffic indicates that all practical means of obtaining maximum stability must be explored if we are to maintain satisfactory service under the increasingly severe traffic on these truck routes.

It is quite possible that the studies now under way may show that greater intial compaction in the pavements being built with penetration grade asphalt in the East is desirable because of currently increasing truck traffic. Some recent pavement failures under heavy truck traffic indicate that compaction and stability requirements that were adequate under the traffic of a few years ago may be inadequate now. If greater initial compaction will help to solve this problem and becomes a general practice, it may well be necessary to adopt kneading compaction or some combination of tamping and compression, such as that used in designing the Soledad-Gonzales mixture for molding laboratory test specimens, in order to obtain densities that will be comparable to such higher pavement densities.

The authors' data are encouraging in their indication that the stabilometer test and the compression test can be correlated and in their corroborative evidence that the volumetric theory of design is, in common with the surface area method, fundamentally sound.

The writer feels that the authors have contributed very materially to the science of mixture design and testing in their objective presentation of their data and their excellent correlation with a successful field project. Further work along these same objective lines will inevitably promote better understanding of the factors that determine pavement behavior and perhaps eventual agreement on the simplest and most dependable sort of an evaluation test.

MR. B. A. VALLERGA AND E. ZUBE (*authors' closure*).—The authors are pleased to have as thorough a discussion of their paper as has been prepared by Mr. Carpenter.

It is evident that Mr. Carpenter approves the approach used by the authors in studying the problem of how to design asphaltic paving mixtures, but that he disagrees somewhat with them in the matter of interpretation of data. After carefully reviewing Mr. Carpenter's remarks, the authors are prepared to stand firm on their initial interpretation as expounded in the original paper and to attribute any variations suggested by Mr. Carpenter to basic differences in viewpoint as to what properties are being evaluated by the various test methods. Therefore, in the interest of brevity, rather than to discuss each contested point individually, the authors will concentrate on exploring the reasons for these basic differences in viewpoint.

Three assumptions are made by Mr. Carpenter which appear to be largely responsible for the differences in interpretation of the data. These are as follows:

1. That the direct compression test is a measure of "stability."
2. That a stability curve must have a

maximum or "peak" value to be valid and this "peak" value must coincide with or be very near the design asphalt content.

3. That the density of the "aggregate portion of the material" correlates with "stability."

The authors would like to restate the above assumptions, in their respective order, to concide with their own beliefs, and include brief supporting statements, as follows:

1. That the direct compression test is *not* a measure of stability.

> The direct compression test is merely a measure of the ability of an asphaltic mix to hold itself together. This cannot be defined as the type of stability required of a mixture to sustain wheel loads on a road. According to this test a cohesionless aggregate has no strength and is therefore unstable, which is not in accordance with the facts. Moreover, as is apparent from Fig. 4, temperature greatly affects the magnitude of the strength value obtained with the direct compression test. This is evidence that the test measures primarily the properties of the asphalt and its effectiveness as a binder. The authors firmly believe that the stability of an asphaltic mix is due primarily to the strength properties of the aggregate and that the direct compression test, *per se*, is wholly inadequate as an *indicator of stability.*

2. A stability curve *need not* have a maximum or "peak" value to be valid, and the "peak" value (if one is present) *need not* coincide with or be very near the design asphalt content.

> On the basis that stability of an asphaltic mixture is primarily a function of aggregate strength, it is evident that an aggregate without any asphalt should have a high stability rating, very nearly the maximum to be expected in the mix. Addition of asphalt generally causes a slight increase in stability because of the more intimate contact obtained between particles through better compaction made possible by the asphalt. However, in many cases because of the lubricating characteristics of the asphalt, no increase of stability is obtained with additions of asphalt to the dry aggregate and, in some cases, any addition of asphalt will cause the aggregate to drop in stability.
>
> Therefore, a characteristic stability curve is one that shows a relatively high value of stability for dry aggregate (actual magnitude of stability depending on strength characteristics of aggregate) followed by a constant value of or a gradual increase or decrease in stability with increasing asphalt content up to the point where the lubricating effect of excessive asphalt film thicknesses causes a rapid drop in stability. In accordance with this hypothesis, it is possible to develop stability curves which have no "peak" value at all over the entire range of asphalt contents normally considered in design. It is also the firm belief of the authors that any "peak" stability values that do develop generally occur at extremely low asphalt contents in comparison with values used in construction (that is, design asphalt contents). Selecting a design asphalt content should, therefore, *not be predicated on maximum stability values but on the premise that for a given aggregate and aggregate gradation as much asphalt should be added to the mix as it will tolerate to secure maximum durability, but not so much as to cause a drop in stability below an established minimum.*
>
> In the use of the stabilometer, for example, less consideration is given to the asphalt content at maximum stability than to the asphalt content be-

yond which there may be the danger of instability. The State of California considers that a minimum stabilometer value of 35 will insure sufficient stability to carry heavy, main-line California traffic.

3. That the density of the "aggregate portion of the material" *does not* necessarily correlate with "stability."

The authors do agree with Mr. Carpenter that aggregate density does correlate with the results obtained from the direct compression test as can be shown from the data in Fig. 4, but for the reasons cited under (1) above, they do not concur with Mr. Carpenter in his belief that the compression test is a stability test. Furthermore, the authors also agree with Mr. Carpenter that there is no correlation between aggregate density and stabilometer values as is fairly obvious from Fig. 5, but unlike Mr. Carpenter the authors consider the stabilometer to be an excellent indicator of stability. In other words, there is no disagreement here in reading the data, but just a difference in opinion as to whether the stabilometer or the direct compression test defines "stability."

It is not possible in this closure to ferret out all the differences in viewpoints. However, it should be evident to the reader that there do exist basic differences in philosophy, principally with respect to the question of what is stability and how it should be measured.

Most test procedures referred to as "stability tests" are really not used as such but merely as tools or yardsticks for establishing design asphalt contents. The direct compression test, the Marshall test, and the Hubbard-Field test fall within this category. Fortunately, if these test methods are correlated with performance and if their testing procedures are followed explicitly, they do yield results that in most cases enable the experienced engineer to arrive at proper values of design asphalt contents, and serviceable pavements have been constructed with their use. Application of these test methods, however, should be limited to the mixes and conditions which prevailed when the method was correlated and they should not be applied indiscriminately.

Without persuing the matter in greater detail, the authors would like to reiterate the conclusions presented in the original paper, with the observation that differences in interpretation of data in connection with asphaltic paving mixtures will always exist as long as there is this lack of agreement on basic premises.

Any discussions of the effects of compaction on stability or density on stability, or the relationship between design asphalt content and stability, should be delayed until the questions of what constitutes stability and how it is measured are resolved.

A COMPARISON OF METHODS OF TESTING FOR DISSOLVED OXYGEN IN WATERS OF HIGH PURITY*

By Walter L. Riedel[1]

Synopsis

An investigation of several methods of testing for dissolved oxygen, in a high purity water, was conducted under the sponsorship of the Dissolved Oxygen Subsection, Subcommittee IV, ASTM Committee D-19 on Industrial Water. The methods studied were the referee and non-referee Methods of D 888[2] and the Schwartz-Gurney B procedure with dead stop, potentiometric, and starch titrations. A deaerated feedwater, low in dissolved oxygen and organic or inorganic materials, was used as the basic sample. Organic and inorganic materials were introduced for positive and negative interferences separately, and an air-saturated solution added to increase the dissolved oxygen content.

A statistical technique, the analysis of variance, was applied to the data collected and furnished a basis for various conclusions as to the precision of the methods.

Many methods have been developed for the determination of dissolved oxygen in industrial water and each of the methods has its advocates. Conflicting opinions exist among the proponents of the more popular methods in regard to the application of some methods to certain types of waters, the accuracy and precision that can be obtained, and the procedure to be used to derive the end point. Recently there has been considerable debate between the advocates of colorimeteric *versus* electrometric methods of determining the end point. Even the proponents of electrometric end point procedures are not in agreement as to whether potentiometric or dead-stop apparatus should be utilized. The evidence therefore indicates the need for a practical comparison of the methods most generally applied in order to evaluate the methods and to explore the variables existing among them—titration procedures, technicians performing the test, and varying types of water at several oxygen levels.

The Heat Exchange Institute recognized the need and carried out an extensive test program in 1945–1946. This program contributed greatly to the available data on the comparative performance of four methods when conducted with potentiometric titration.

The Dissolved Oxygen Subsection, Subcommittee IV on Methods of Analysis of ASTM Committee D-19 on Industrial Water, desired to investigate the variables existing between methods when conducted with potentiometric, dead-stop, and starch titration procedures, different technicians, and varying levels of oxygen in relatively pure water and water containing oxidizing or reducing

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Engineer, Consolidated Gas Electric Light and Power Co., Baltimore, Md.

[2] Tentative Methods of Test for Dissolved Oxygen in Industrial Water (D 888 – 49 T), 1952 Book of ASTM Standards, Part 7, p. 1155.

interference. They appointed a task group to conduct a practical test program in Baltimore, Md., from July 14 to 18, 1952, the data obtained to serve as a basis for revision of ASTM Method D 888.[2]

TEST PROGRAM

Improved techniques and the availability of less expensive electrometric apparatus have resulted in increased application of potentiometric and dead-stop end point procedures. The Schwartz-Gurney B Method, because it compensates directly for the oxygen in the reagents, has been widely used with the Daugherty sampling modification for starch end point determinations and has also been adapted to potentiometric and dead-stop end point apparatus. The task group chose, therefore, to compare the ASTM Method D 888 with the Schwartz-Gurney B Method, using three end point procedures.

The six procedures selected for comparison were assigned the first six Roman numbers as follows:

Method I. Referee Method of ASTM Method D 888.[2]

Method II. Schwartz-Gurney Procedure B with potentiometric end point (**1**).[3]

Method III. Schwartz-Gurney Procedure B with dead-stop end point (**2**).

Method IV. Schwartz-Gurney Procedure B with starch end point (**3**, **4**).

Method V. Non-Referee Method A of ASTM Method D 888.

Method VI. Non-Referee Method B of ASTM Method D 888.

The Daugherty sampling modification was used in all the Schwartz-Gurney procedures.

These methods were performed by five operators; the Referee Method and Non-Referee Method A were performed simultaneously as Method I by the same operator. Various companies interested in the field of dissolved oxygen testing supplied the personnel for the test program. All operators had had previous experience in dissolved oxygen test work and were familiar with one or more of the six procedures, but none had previously executed all of them, thus lending practicality to the program. A day was spent in familiarizing them with the different methods, the apparatus, and the sampling setup.

The group was supervised by a chemical engineer. A statistician attended the entire program to insure that the statistical plan was correctly carried out and to furnish professional assistance in making whatever changes in the prearranged program which might be necessary after the program had begun.

Four concentrations were used to explore the effects of dissolved oxygen content and positive or negative interferences. Concentration A was mechanically deaerated water of high purity, containing approximately 0.0000 to 0.0050 ppm of dissolved oxygen. All other concentrations were prepared by admixture with this basic sample. Concentration B was a water containing approximately 0.040 to 0.060 ppm of dissolved oxygen. Concentration C contained approximately 2 ppm of tannin and 0.2 ppm of sodium sulfite as SO_3 ion reducing interference. For concentration D, 0.2 ppm of NO_3 ion as sodium nitrate oxidizing interference was added to the basic sample. It was assumed that the basic sample was saturated with ferric ion.

Preparations:

The water for the basic sample was taken from the outlet of a tray-type deaerating heater, operating at full capacity on condensate from a steam turbine.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1119.

supplied with steam from a 580,000 lb per hr boiler. Evaporated Baltimore City water was used as make-up to the condensate cycle. The water for all samples originated at the outlet of this deaerator located approximately 100 ft above the room where all sample collecting, fixing, and titrating were performed. The sample was conducted through copper tubing from its source to the sample table as shown in Fig. 1.

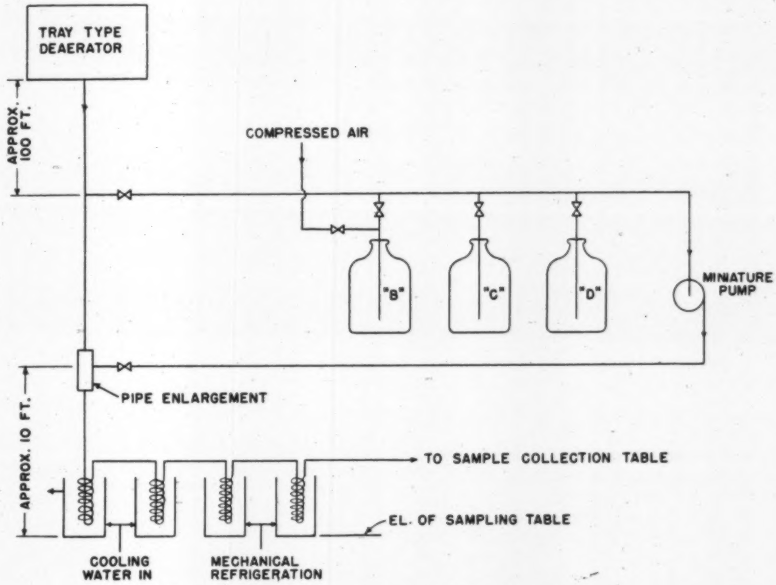

FIG. 1.—Schematic Drawing of Sampling System.

Air-saturated water for concentration B and solutions of the interference materials for concentrations C and D were made up and stored in 5-gal pyrex bottles, located just above the test location. The bottles contained, respectively, air-saturated water, quebracho tannin and sodium sulfite solution, and sodium nitrate solution. Provision was made for pumping from one of these bottles into the basic sample stream when the corresponding concentration was desired. A miniature, adjustable stroke, chemical pump took suction from a manifold connecting the bottles and discharged to the line from the deaerating heater. This permitted controlled volumes of the B, C, or D solutions to be introduced into the sample.

An enlarged pipe section was placed in the sample line at the discharge from the chemical pump. This fitting was fabricated from a 1-ft section of 1-in. brass pipe so designed as to permit the $\frac{1}{4}$-in. copper tubing pump discharge line to enter the 1-in. brass pipe tangentially near its inlet end. The rest of the sample line was fabricated of $\frac{3}{8}$-in. copper tubing.

The sample then ran through four copper cooling coils in series (approximately 200 ft of copper tubing), two of which were cooled with water and two by a mechanical refrigeration system, before going to the sample table. The arrangement of the sampling table is shown in Fig. 2. The sample line entered the vertical axis of a hub. From this hub the stream was discharged through five identical horizontal taps, each 4 ft in length. A valve and hose nipple were placed at the end of each of these split sample lines. The sample apparatus used for each individual method was connected with rubber tubing to the hose nipples provided.

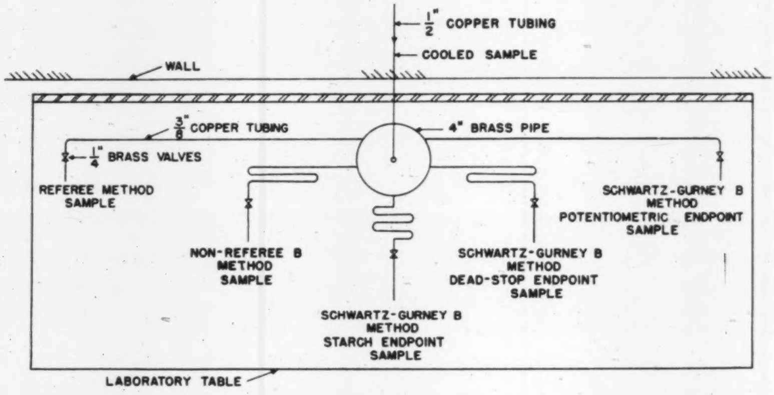
1/2" COPPER TUBING
COOLED SAMPLE
WALL
3/8" COPPER TUBING
4" BRASS PIPE
1/4" BRASS VALVES
REFEREE METHOD SAMPLE
SCHWARTZ-GURNEY B METHOD POTENTIOMETRIC ENDPOINT SAMPLE
NON-REFEREE B METHOD SAMPLE
SCHWARTZ-GURNEY B METHOD DEAD-STOP ENDPOINT SAMPLE
SCHWARTZ-GURNEY B METHOD STARCH ENDPOINT SAMPLE
LABORATORY TABLE

FIG. 2.—Arrangement of Sample Lines at Sample Collection Table.

The test agenda was divided into two parts or replications, each consisting of 20 tests or rounds. A Latin-Square statistical plan was used. Table I illustrates the scheme for concentration A, first replication.

TABLE I.—OPERATOR METHOD SCHEDULE, CONCENTRATION A, FIRST REPLICATION.
Operator identification by numbers 1 to 5 inclusive.

Round No.	ASTM Method D 888		Schwartz-Gurney B Method		
	Referee Method	Non-Referee B Method	Potentiometric	Starch	Dead-Stop
2	1	4	3	2	5
17	2	5	4	3	1
18	4	2	1	5	3
19	5	3	2	1	4
22	3	1	5	4	2

A study of this table reveals the complete balance that was obtained. Thus, in five rounds each operator used each method. The other concentrations had similar Latin Squares, each being selected randomly. The order of running of the rounds also was selected randomly. Randomness was used to enlarge the scope of the test and to reduce any bias due to time. This randomness was restricted in the first replication in that two or more consecutive rounds were tested at the same concentration. Since it was found necessary to wait no longer than 15 min for stabilization of the sample after changing the concentration, this restriction was removed in the second replication.

Apparatus:

The Referee Method (ASTM D 888) samples were collected in calibrated 500-ml McLean sampling tubes as modified by the Engineering Experiment Station, U. S. Navy, and the Heat Exchange Institute and described in ASTM Method D 888. Samples for the Schwartz-Gurney B procedures were collected in glass bottles with conical-tip glass stoppers. Their capacities were adjusted by the addition of plastic rods, so that they would hold 500 ± 1.0 ml or 250 ± 0.5 ml. The Non-Referee Method B sample of ASTM D 888 was collected in a 300-ml B.O.D. bottle having a raised lip around the neck and a glass stopper ground to a conical lower tip. The capacity of the B.O.D. bottle was measured to the nearest milliliter. A single glass sampling tube slightly longer than the depth of the bottle was used to introduce the sample into the bottle. All glass stoppers were tied to the necks of the bottles with loops of cord.

The Schwartz-Gurney B Method samples were split with stainless steel manifolds constructed so that two of the arms delivered volumes of water in the ratio of one to two with the arm to the 500-ml bottle. The sample bottle containers were so designed that the top of the bottle necks were more than an inch below the overflowing water.

The room in which all tests were performed, from sample collection to titration, was illuminated with fluorescent lighting.

Microburets were used in all titrations; but, due to the varied sources from which the apparatus was derived, the type of burets used and the smallest subdivision on them were not identical in all methods. Differences are described later.

Glass electrode pH meters were utilized for the millivoltage measurements in the potentiometric methods. The potentiometric procedures differed only in the sampling system and strength of the thiosulfate used for titration.

The apparatus used in the dead-stop titration procedure was an adaptation of the apparatus of Ulmer, Reynar, and Decker (2). According to the procedure followed, the end point was always approached from the reducing side. Microburets with the smallest subdivision of 0.05 ml were used for the dead-stop and potentiometric titrations of the Schwartz-Gurney B Method, while the microburets used in the other methods were subdivided to 0.01 ml. Since high precision was obtained in the dead-stop titration, this difference in apparatus is not considered significant.

All apparatus and reagents were as specified in ASTM Method D 888 and the second draft of the Proposed Revision to Non-Referee Method A of D 888 (see Appendix), except as indicated in the text of this paper.

Sufficient quantities of all reagents required were made up the week previous to the start of the program. The dilute sodium thiosulfate and potassium iodate solutions were made by dilution from 0.05 *N* stock solutions daily prior to the initial test round. A fresh supply of all other reagents was acquired daily from the stock supply prior to the initial round. Sufficient 0.1 *N* iodine was added daily to the alkaline iodide solution used in the referee method to react with the reducing interference in the samples.

Performance:

Concentration A water ran continuously through the sampling system from 48 hr before the first samples were collected until the samples for the final round of the program were completed. Whenever it was desired to change to another concentration, the proper valve changes were made and the stroke of the miniature chemical pump was adjusted. (The stroke adjustment required to deliver the necessary volume for each concentration had been predetermined.)

Such valve changes and pump adjustments were made immediately after the samples for the previous round had been collected to permit time for stabilization.

The sample flow rates were adjusted to 1000 ml per min at the start of each day's test period and checked at random daily to maintain this rate throughout the test period. Sample temperatures varied from 75 to 78 F daily, a minimum of 5 F below room temperature.

The samples for each round were collected for a minimum of 20 min during the first series of 20 rounds and for a minimum of 15 min during the second replication. Each round of samples were drawn simultaneously by the operators and, in addition, each round was completed by all operators before the next sample collection period was started.

TABLE II.—SUMMARY OF TEST DATA.

	ASTM D 888		Schwartz-Gurney B		
	Referee	Non-Referee B	Potentiometric	Dead-Stop	Starch
			CONCENTRATION A		
First Replicate	0.0097 a[a]	0.0451 d[b]	0.0051 c	0.0000 b	0.0083 e
	0.0050 b[b]	0.0320 e	0.0013 d	0.0058 c[b]	0.0109 a[b]
	0.0047 d	[c] b	0.0058 a[b]	0.0000 e	0.0051 c
	0.0036 e	0.0178 c	0.0032 b	0.0032 a	0.0051 d
	0.0036 c	0.0084 a	−0.0038 e	0.0000 d	0.0045 b
Second Replicate	0.0097 d	0.0184 c	0.0134 e	0.0019 a	0.0064 b
	0.0013 e	0.0393 d	0.0026 a	0.0032 b	0.0026 c
	0.0038 c	[c] b	0.0006 d	0.0064 e	0.0038 a
	0.0074 b	[c] a	0.0064 c	0.0013 d	0.0006 e
	0.0075 a	0.0236 e	−0.0019 b	0.0006 c	−0.0064 d
			CONCENTRATION B		
First Replicate	0.0668 c	0.0813 a	0.0685 e	0.0672 b	0.0659 d
	0.0667 a	0.0876 e	0.0672 c	0.0685 d	0.0749 b
	0.0623 b[b]	0.0866 c	0.0717 d	0.0640 a	0.0666 e
	0.0684 d	0.0818 b	0.0595 a[b]	0.0608 e	0.0672 c
	0.0700 e	0.0997 d[b]	0.0704 b	0.0691 c[b]	0.0730 a[b]
Second Replicate	0.0654 b	0.0939 a	0.0659 c	0.0640 d	0.0698 e
	0.0759 e	0.0902 c	0.0736 b	0.0717 a	0.0800 d
	0.0646 a	0.0829 e	0.0646 d	0.0646 c	0.0691 b
	0.0631 d	0.0908 b	0.0608 a	0.0608 e	0.0582 c
	0.0676 c	0.0892 d	0.0582 e	0.0640 b	0.0730 a
			CONCENTRATION C		
First Replicate	0.0030 e	0.0357 d[b]	−0.0128 a[b]	0.0237 c[b]	−0.0077 b
	0.0014 d	0.0052 c	0.0032 b	0.0006 e	[c b a] a
	0.0054 a	[c] e	0.0000 c	0.0064 b	−0.0045 d
	0.0076 c	[c] b	−0.0032 d	0.0032 a	−0.0006 e
	−0.0015 b[b]	[c] a	0.0058 e	0.0045 d	0.0000 c
Second Replicate	0.0039 d	[c] a	0.0038 b	0.0000 e	0.0038 c
	0.0041 c	[c] b	−0.0013 a	0.0006 d	0.0006 e
	0.0034 b	0.0210 d	0.0096 e	0.0019 c	−0.0224 a
	0.0015 e	[c] c	−0.0006 d	0.0000 a	−0.0006 b
	0.0025 a	[c] e	0.0032 c	−0.0032 b	0.0064 d
			CONCENTRATION D		
First Replicate	0.0042 b[b]	0.0304 c	0.0141 e	−0.0013 d	0.0160 a[b]
	0.0090 e	0.0666 d[b]	−0.0173 a[b]	0.0154 c[b]	0.0070 b
	0.0151 a	0.0346 e	0.0134 c	0.0128 b	0.0141 d
	0.0101 c	0.0346 b	0.0064 d	0.0128 a	0.0077 e
	0.0128 d	0.0409 a	0.0032 b	0.0048 e	0.0077 c
Second Replicate	0.0120 b	0.0367 d	0.0154 e	0.0032 c	0.0051 a
	0.0112 e	0.0304 c	0.0083 d	0.0096 a	0.0045 b
	0.0185 d	0.0357 a	0.0224 b	0.0141 e	0.0365 c
	0.0135 c	0.0409 e	0.0262 a	0.0128 b	0.0224 d
	0.0042 a	0.0341 b	0.0122 c	0.0064 d	0.0102 e

[a] Operator identification indicated by letters a to e inclusive.
[b] Operator was found statistically different from others with this method in first replication.
[c] No starch color developed.

Just prior to the starting period for a round, data sheets, indicating the method assigned to each operator for that particular round, were distributed by the statistician. Then at a signal from the supervisor of the test, all samples were withdrawn simultaneously, immediately fixed, and then titrated according to the procedures of the various methods.

Two milliliters of each of the fixing reagents were used in all methods but the dead-stop titration procedure. In this procedure the volumes of the manganous sulfate and alkaline iodide fixing reagents were reduced to 1 ml, and 3 ml of concentrated hydrochloric acid was substituted for sulfuric acid.

STATISTICAL ANALYSIS

The analysis of variance was applied to the data obtained from the more than 200 individual determinations of dissolved oxygen. Detailed computations are not included because of their bulk, but the original data are shown in Table II for any desired corroborative calculations.

The values shown in Table II are dissolved oxygen concentrations in parts per million as measured and calculated by the respective procedures. They are grouped primarily by concentrations and secondly in chronological order. Each line in the table was obtained by concurrent determinations on simultaneously drawn, identical samples. The operator who made each determination is identified by the adjoining lower-case letter (a to e, inclusive). From this presentation an evaluation of any of the prime variables—method, operator, and concentration—can be made. Study of suspected additional variables also is possible.

Extensive analyses of variance disclosed a number of significant relationships among the variables under study. Those concerning the three prime variables which the program was designed to evaluate are discussed first, followed by a few related conclusions of interest.

There were significant differences among the results by the several operators during the first replication. Those by four of the five operators departed from the self-established norm by more than random dispersion. One obtained low results with the Referee Method. Another got low results with the Schwartz-Gurney Procedure B potentiometric end point and high ones with the Schwartz-Gurney B starch end point. Two others turned in high results with the Schwartz-Gurney Procedure B with dead-stop end point and Non-Referee Method A with starch end point, respectively. These differences did not persist in the second replication so that they may be attributed to operator inexperience with specific procedures. In fact, the operators were statistically homogeneous in the second replication and may be considered so for the program as a whole.

The four concentrations were calculatedly different so that it was perhaps superfluous to demonstrate that difference statistically. Although the several methods did not agree in the amount of dissolved oxygen present, they were consistent in measuring the difference between concentrations A and B. This consistency disappeared with the presence of interference (concentrations C and D). The methods could not agree whether concentration C was greater or less than concentration A. The Schwartz-Gurney Procedure B with dead-stop end point yielded higher results in one replication and lower in the other for an average indicating that concentration C is the greater, which is theoretically correct. All agreed that concentration D was greater than concentration A, but there was little unanimity on the degree of this difference.

The operators having been established as a homogeneous group, and the concentrations as significantly different, it was possible to measure statistical similarities among the methods. No absolute standard of dissolved-oxygen content was available in the program so that judgment of only precision, not accuracy, could be derived.

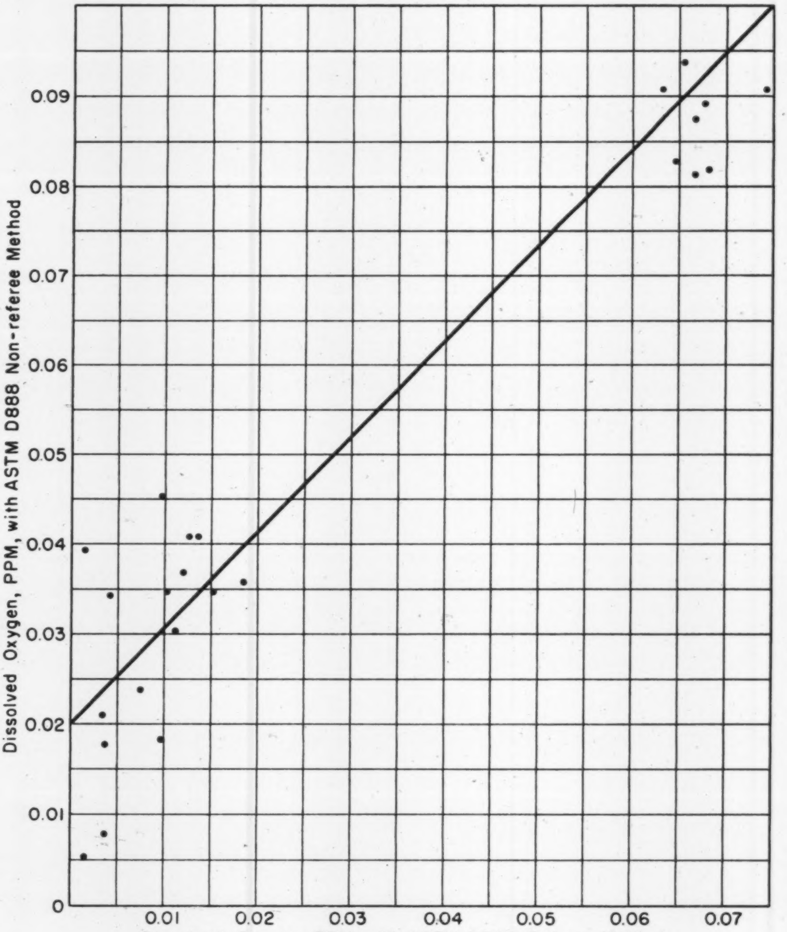

FIG. 3.—Comparison of ASTM Methods D 888, Referee and Non-Referee B Methods.

The best indication of relative precision is given by the over-all standard deviation for each method. Analysis of Variance yields such values free from operator and concentration effects. These standard deviations for the five methods yielding complete data in both replications are tabulated below:

Referee Method of ASTM D 888	0.0036 ppm
Schwartz-Gurney Procedure B with dead-stop end point	0.0045 ppm
Non-Referee Method B of ASTM D 888	0.0069 ppm
Schwartz-Gurney Procedure B with potentiometric end point	0.0072 ppm
Schwartz-Gurney Procedure B with starch end point	0.0080 ppm

Although these show Method I to be most precise, the difference of 0.0009 in standard deviation between the Referee

Method and the Schwartz-Gurney Procedure B with dead-stop end point is scarcely significant. These two methods thus should be considered of about equal precision. Similarly, the other three methods should be grouped together as having lesser precision but being similar among themselves.

Undoubtedly there was some increase in these deviations because of the changes in concentration and some changes in conditions with time so that they should not be used as a criterion of reproducibility for successive determinations from a single sampling location. In the course of the program, a few consecutive rounds were made without change in concentration. Presumably reproducibility on such successive pairs of identical samples should be better and possibly more indicative of normal applications. Accordingly the results from all such pairs were abstracted and analyzed for method standard deviation, with the following results:

Referee Method of ASTM D 888	0.0026 ppm
Schwartz-Gurney Procedure B with potentiometric end point	0.0043 ppm
Schwartz-Gurney Procedure B with dead-stop end point	0.0044 ppm
Schwartz-Gurney Procedure B with starch end point	0.0051 ppm
Non-Referee Method B of ASTM D 888	0.0060 ppm

All standard deviations decreased, as expected, but the Referee Method is still the most precise with the remaining four approximately equivalent to each other.

Although the Non-Referee B Method is in the same precision range as a majority of the procedures, the level of results by it was distinctly higher. This is illustrated by Fig. 3 in which all results by this method are plotted against those by the Referee Method. (Statistically aberrant results have been excluded.) The least-squares best fit gave a straight line with an intercept of 0.02 ppm and a slope of 1.0. This means that results by the Non-Referee B Method averaged 0.02 ppm greater than those by the Referee Method.

Data for the Non-Referee A Method were so incomplete that they are not included in Table II. Starch gave end points and the method produced results only for concentrations A and B in the second replication. Analysis of these limited data showed that the Non-Referee A Method gave values with concentration A that were higher (borderline significance) than those by the Referee Method or any of the Schwartz-Gurney procedures. With concentration B, the Non-Referee A Method was in the same range as these methods. Its standard deviation was 0.0078 ppm, in the lower precision group.

COMPARISON OF PROCEDURES

Essentially two methods of sampling were used, the McLean sampling tube and the Daugherty sampling modification of the Schwartz-Gurney B Method. Since the sampling technique is fundamental to any test, the sampling system used has a great influence on the results obtained.

Previous investigations (6) have indicated that double-size samples are required to obtain the same degree of precision with the Schwartz-Gurney B Procedure as with a 500-ml sample. If the sample size were the only consideration entering into the precision, this would be important. However, a system that automatically compensates for the dissolved oxygen in the reagents and interference is desirable. The reduction in precision expected as a result of a smaller sample size was not apparent in the dead-stop titration in this investigation.

For precise work the use of the sampling tube is more desirable. The manipulative skill required in handling this type tube during the sampling and fixing operations is greater than with bottles, and it is harder to keep sample tubes clean. When using sampling tubes with glass stopcocks, which require lubrication, care must be exercised to keep the tubes clean. Teflon stopcocks, which obviate lubrication and a possible source of error, are now available for use with this type sampling tube. Fixing procedures are not difficult and only average technique is sufficient to insure excellent results.

Potentiometric titrations are more time consuming than starch or dead-stop titrations. It is desirable in potentiometric work to have a two-man team to conduct the titration, one to perform the actual titration and the other to record the data. This procedure was used in this program. Even with this assistance the potentiometric titrations required more time than the other methods of titration.

The dead-stop titration procedure is not difficult to perform and can be completed almost as rapidly as a starch titration. The end point is sharp and definite and can be approached by a continuous titration. It is necessary to record only the total amounts of thiosulfate and potassium iodate consumed in the titration.

In the starch titration procedures, the starch color developed in all but one of the samples taken by the Daugherty modification of the Schwartz-Gurney B Method, but it failed to develop in ten samples in the Non-Referee B Method. Seven of the Non-Referee B samples in which insufficient starch color was encountered were samples containing reducing interference.

The starch titration was quite generally known by all the operators who took part in the program. The titrations were made with microburets in color-matched bowls. The sample temperature was high for starch titrations. This resulted from the high rate of sample flow required for simultaneous sampling of all methods and high ambient temperatures. To attempt to cool the samples after fixing would have resulted in an unreasonable delay in the program. The procedure, although carried out in a well lighted room, is always subject to observer error and temperature effects which make the method impractical as compared to electrometric methods which are not subject to either error.

An attempt to perform simultaneously the Non-Referee A Method by the addition of starch to the Referee Method samples was unsuccessful because the color failed to develop in a large number of the samples. Several attempts were made at increasing the amount of iodine in the alkaline iodide, but this did not effect a change in the starch-color production.

The Non-Referee B Method was carried out as prescribed in ASTM Method D 888, with the exception that 0.002 *N* thiosulfate was used, the same as in the Schwartz-Gurney starch titration procedure. The reason for the use of a more dilute solution was to permit comparison of the starch methods with equivalent titrating reagents. The only differences in procedures then were the sampling method and a slight difference in sample size. The precision obtained was greater than the potentiometric or starch titrations of the Schwartz-Gurney procedure. However, accuracy apparently was not obtained as the values were approximately 0.020 ppm high for all concentrations.

It is probable that collection of samples in B.O.D. bottles results in atmospheric contamination during fixing. Under-water sample collecting and fixing should improve the results by this method. The difficulty which was experienced with starch color development is attributed to reducing interferences for which the procedure does not compensate. The difficulty was experienced only with concentrations A and C.

A total of nine rounds had to be repeated, four in the first replication and five in the second. This was due to faulty operation of apparatus or operator procedure. The data of each method from these discarded rounds were destroyed before any calculations were made.

Some further information on the practical usefulness of the several methods can be obtained by studying the summary of results in the light of the foregoing discussion. Table III is such a summary, giving the mean of all dissolved-oxygen determinations by method and concentration.

TABLE III.—AVERAGE (n = 10) DISSOLVED OXYGEN CONTENT (PPM) BY CONCENTRATION AND METHOD.

Concentration	ASTM Method D 888		Schwartz-Gurney B Method		
	Referee Method	Non-Referee B Method	Potentiometric	Starch	Dead-Stop
A..........	0.0056	0.0264	0.0033	0.0041	0.0020
B..........	0.0671	0.0884	0.0660	0.0698	0.0655
C..........	0.0031	[a]	0.0008	−0.0028	0.0038
D..........	0.0111	0.0385	0.0104	0.0131	0.0091

[a] Excluded due to lack of color in seven readings.

Excluding the results by the Non-Referee B Method, which are 0.02 ppm high, those by the other four methods are statistically alike at each concentration. The superior precision of the Referee Method and the Schwartz-Gurney B Method with dead-stop titration, however, encourages a detailed comparison of their performance. There is a practical suspicion that results by the Referee Method tend to be higher than those by the Schwartz-Gurney B Method with dead-stop titration despite the lack of statistical support. However, the differences between average results by the two methods at concentrations A, B, and D are 0.0036, 0.0016, and 0.0020 ppm. This consistent difference makes it surprising that at concentration C, the Referee Method reports not only 0.0007 ppm less than the Schwartz-Gurney B Method with dead-stop titration but 0.0025 ppm less than it does for concentration A. Since the solution of reducing salts for concentration C was prepared in an unstoppered bottle and therefore contained dissolved oxygen, the oxygen content of concentration C must have been greater than that of concentration A; possibly it was of the same level as concentration D. Only the dead-stop titration procedure reflected any increase between concentrations A and C, and this only in the second replication although the average for both replications was slightly higher.

The choice of the most reliable method lies between the Referee Method and the dead-stop titration procedure of the Schwartz-Gurney B Method. A choice cannot be made, however, until sound information on accuracy, as well as precision, is available.

SIGNIFICANCE OF DATA

A comparison of the methods indicated that precise results can be obtained with random sampling of industrial water, either pure or contaminated, containing low or high levels of dissolved oxygen when the Referee Method of ASTM Method D 888 or the proposed Non-Referee A Method with dead-stop end point titration (see Appendix) is employed. The precision of both of these methods was far superior to that obtained with the starch end point titrations or the Schwartz-Gurney B Method with potentiometric titration. For control purposes or where the highest precision is not required, the Schwartz-Gurney B Method with potentiometric or starch end point determination gives sufficiently precise results.

The fact that operators can be trained

in a reasonably short time to perform any of the procedures studied and then obtain precise results is evident. Some differences occurred in the first replication, but in the second replication the operators were statistically equal.

No statement as to the accuracy of any method can be made as the result of this program.

As has been previously shown, accuracy and precision cannot be obtained with a starch-titration procedure unless a large number of tests are performed and a correction for the starch end point based upon sensitivity at the temperature of titration applied. When considerable reducing substances are present in the water, the starch titration is not applicable in methods that do not compensate for interference.

RECOMMENDATIONS FOR FURTHER WORK

Although this program has established the precisions obtainable with the methods investigated, another test program to evaluate the accuracy of the methods is indicated.

A comparison of the relative accuracies and precision of the Referee Method and Schwartz-Gurney B procedure, using potentiometric and dead-stop end points and sampling tubes for the collection of all samples, would be especially valuable.

Acknowledgments:

Since this is the report of a group effort, acknowledgment should be made to the other contributors and participants. These include:

Mr. T. H. Daugherty, Chairman of the Dissolved Oxygen Subsection, Subcommittee IV of ASTM Committee D-19 on Industrial Water, and his committee members who sponsored the project;

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APPENDIX

TESTS FOR DISSOLVED OXYGEN IN INDUSTRIAL WATERS—PROPOSED REVISION TO ASTM D 888 - 49 T,[2] NON-REFEREE METHOD A[4]

Application

10. This method, based upon the Schwartz-Gurney B Method,[5] is applicable to the determination of dissolved oxygen in industrial water when a simple, routine, and precise method is required.

Apparatus

11. The following apparatus is required:

(*a*) *Bottle.*—One approximately 500-ml glass bottle with a conical-tip glass stopper. Label as Bottle A.

NOTE 15.—Adjust the volume of this bottle by adding glass marbles or other noninterfering filler, so that it will hold 500 ± 1.0 ml of water at room temperature when completely filled and stoppered.

(*b*) *Bottles.*—Two approximately 250-ml glass bottles with conical-tip glass stoppers. Label as Bottles B and C.

NOTE 16.—Same as Note 15 above, except adjust volumes to 250 ml ± 0.5 ml.

(*c*) *Manifold, Sampling.*—A stainless steel, copper, or glass manifold, similar to that shown in Figs. 4 and 5, constructed so that two of the exit arms each deliver volumes of water in the ratio of 1 to 2 with the exit arm to the 500-ml bottle.

(*d*) *Container, Sampling.*—A suitable container, such as shown in Fig. 4, with an internal support block, both preferably constructed of noncorroding metal and of such dimensions that the three sampling bottles, with the sampling manifold attached, will conveniently fit with the top of the bottle necks at least 1 in. below the overflowing water level.

(*e*) *Dishes, Porcelain.*—Two porcelain dishes of at least 600-ml capacity and of matched whiteness.

(*f*) *Miscellaneous.*—Burets and pipets as described in Section 5. The motor stirrer described in Section 5(*d*) is convenient.

Reagents

12. (*a*) *Alkaline Potassium Iodide Solution.*—Same as Section 6(*a*).

(*b*) *Manganous Sulfate Solution.*—Same as Section 6(*d*).

(*c*) *Sulfuric Acid Solution.*—Same as Section 6(*e*).

(*d*) *Hydrochloric Acid*, concentrated, reagent grade.

(*e*) *Sodium Thiosulfate Solution* (*0.05 N*).—Same as Section 6(*f*).

(*f*) *Sodium Thiosulfate Solution* (*0.002 N*).—With a calibrated pipet, transfer 10.00 ml of the 0.05 *N* $Na_2S_2O_3$ solution (Section 12 (*e*)) to a 250-ml volumetric flask. Dilute to the mark with distilled water and mix completely. This solution shall be prepared not more than 12 to 15 hr before using.

(*g*) *Potassium Iodate Solution* (*0.002 N*).—First prepare a 0.1 *N* solution by drying reagent grade KIO_3 at 120 C, dissolving 3.5669 g in distilled water, and diluting in a volumetric flask with distilled water to 1 liter. Dilute 2.00 ml of this 0.1 *N* solution with distilled water in a volumetric flask to 100 ml to prepare 0.002 *N* solution. This solution shall be prepared not more than 12 to 15 hr before using.

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[4] To replace present Non-Referee Method A, which then becomes Non-Referee Method B, with appropriate renumbering of paragraphs and notes.

[5] M. C. Schwartz and W. B. Gurney, "The Determination of Traces of Dissolved Oxygen by the Winkler Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, pp. 796–820 (1934).

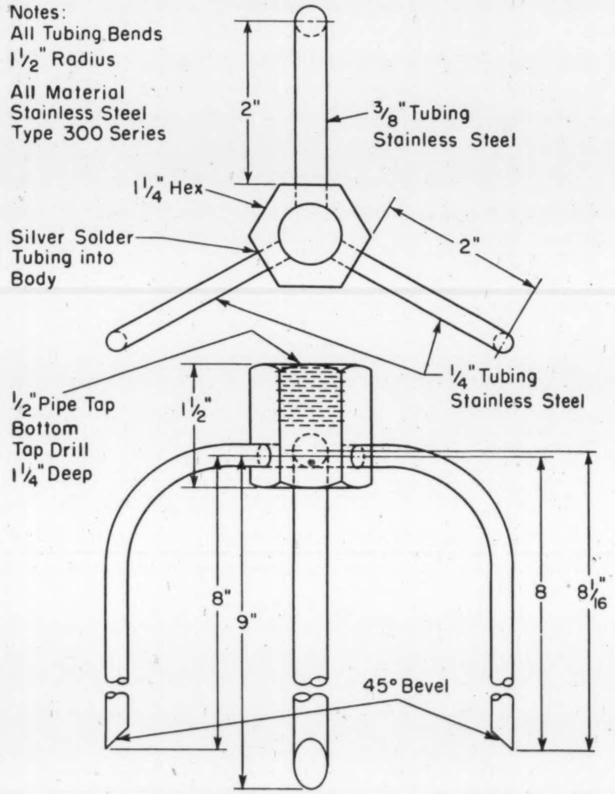
Notes:
All Tubing Bends
1 1/2" Radius
All Material
Stainless Steel
Type 300 Series
2"
3/8" Tubing
Stainless Steel
1 1/4" Hex
Silver Solder
Tubing into
Body
2"
1/4" Tubing
Stainless Steel
1/2" Pipe Tap
Bottom
Tap Drill
1 1/4" Deep
1 1/2"
8"
9"
8
8 1/16"
45° Bevel

FIG. 4.—Three-Arm Manifold for Dissolved Oxygen Test, ASTM Methods D 888, Non-Referee Method A.

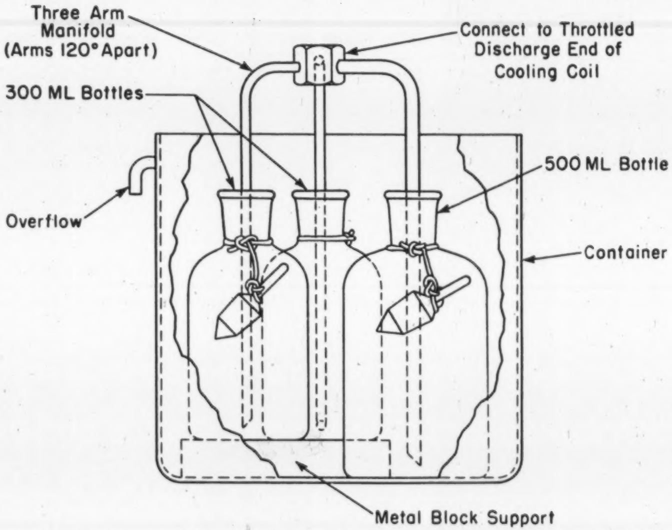
Three Arm
Manifold
(Arms 120° Apart)
Connect to Throttled
Discharge End of
Cooling Coil
300 ML Bottles
500 ML Bottle
Overflow
Container
Metal Block Support

FIG. 5.—Sampling Assembly for Dissolved Oxygen Test, ASTM Methods D 888, Non-Referee Method A.

NOTE 17.—For starch and potentiometric titrations, the KIO_3 solution need not be exactly 0.002 *N*, since the same amount of solution is added to each of the two samples to be titrated, whereby the equivalent titration value of the KIO_3 cancels in the subtraction of the two titration values. However, the 0.1 *N* KIO_3, if carefully prepared from the pure salt, is useful for standardizing the 0.05 *N* $Na_2S_2O_3$ solution (Section 12(*e*)).

(*h*) *Stable Starch Solution.*—Make a paste of distilled water and 6 g of potato or arrowroot starch and dilute with distilled water to 1 liter. Add 20 g of reagent grade KOH and stir until dissolved. Allow to stand for 2 hr and add 27.5 ml of reagent grade HCl (sp gr 1.19) and 6 ml of glacial acetic acid. Adjust pH with HCl or KOH additions as required to approximately pH 4.

Procedure

13. (*a*) *Sampling.*—Arrange the volume-adjusted 500-ml sample bottle and the two volume-adjusted 250-ml sample bottles in the sampling container as shown in Fig. 5. Connect the inlet end of the sampling manifold to the sampling line with copper or rubber tubing.

NOTE 18.—The sampling line shall contain a suitable cooling coil if the water being sampled is above room temperature, in which case the sample shall be cooled to within 5 F of room temperature. If a cooling coil is used, the valve for cooling water adjustment shall be at the inlet to the cooling coil and the overflow shall be to a point of lower elevation. The valve for adjusting the flow of sample shall be at the outlet from the cooling coil.

Lower the sampling manifold into the bottles with the largest diameter arm in the 500-ml bottle, bottle A. Open the valve on the sampling line to provide a flow rate of approximately 1 liter per minute and flush bottles and container with warm (approximately 120 F) sample to expel atmospheric oxygen, allowing the container to overflow at least five times its volume. Adjust the rate of flow of cooling water until the temperature of the sample is within 5 F of room temperature. For the first sample, or for the first sample taken after a cessation of sample flow, the sampling line should be allowed to flow for an adequate period, usually at least 20 min, to insure that a representative sample is obtained. Subsequent samples taken without cessation of sample flow can be obtained after flushing the sample bottles six to ten volume changes. Without stopping or throttling the flow of sample, carefully raise the manifold until the arms are out of the bottles but still beneath the surface of water in the sampling container, turn the manifold about 60 deg, and lower the arms into the sampling container. Stopper each bottle under water and examine each for entrained air bubbles and for suspended material. If any air bubbles are found, empty all bottles and resample until no air bubbles can be observed. If suspended material such as iron oxide is present, insert suitable filter in the sampling line between the cooling coil and the sampling manifold and resample. The samples are now ready for fixing.

(*b*) *Fixing.*—Remove bottle C. Fix only bottles A and B; *add no reagents to bottle C.* Fill a 2-ml transfer pipet with alkaline potassium iodide solution (Section 12(*a*)) and, with the pipet filled to the tip so that no bubble of air will be forced into the sample, ease the stopper of bottle A out of its seat and simultaneously thrust the pipet tip past it and below the neck of the bottle. Allow the contents of the pipet to drain into the bottle and, as the level in the pipet approaches that of the water in the sampling container, slowly raise the pipet out of the bottle and let the stopper fall back into its seat. In a similar manner, add the same amount of this reagent to bottle B. Fill another 2-ml transfer pipet with manganous sulfite solution (Section 12 (*b*)) and in a similar manner add it to bottle A. In a similar manner, add the same amount to bottle B. Shake and rotate bottles A and B, with stoppers firmly seated, until the manganous hydroxide precipitates are thoroughly mixed throughout the samples. Replace in the sampling container (with sample water still flowing from the manifold arms) and allow to stand until the precipitates have settled below the shoulders of the bottles. Then add from a third 2-ml transfer pipet 2 ml of sulfuric acid solution (Section 12 (*c*)) in the same manner as before to bottle A and to bottle B. Shake and rotate bottles A and B until all precipitate has dissolved. The two simultaneous samples are now fixed and ready for titration.

NOTE 19.—25-ml burets with 5-in. tips have been found to be satisfactory, provided that no lubricant is used on stopcocks, in place of transfer pipets.

(*c*) *Titrating* (*Starch End Point*).—Remove stoppered bottles A, B, and C from the sampling container. Cool all samples to such a temperature that the titrations can be completed at or below 70 F. Pour all the cool solution from bottle A into a porcelain dish marked A, retaining the filler material in the bottle. Rinse the inside of the bottle with about 10 ml distilled water and pour this liquid into dish A. Pour all the cool solution from bottles B and C into a porcelain dish marked B, retaining the filler material in the bottles. Rinse the inside of bottle B with about 10 ml distilled water and pour this liquid into dish B. Add approximately 3 ml stable starch solution (Section 12(*h*)) to each of dishes A and B. If the solutions in *both* dish A and dish B show a blue color, proceed with the titration. If one or both of these samples does not show a blue color after the addition of starch solution, add 0.002 *N* potassium iodate solution (Section 12(*g*)) in increments of 0.1 ml to the colorless sample until it just shows a blue color. Then add exactly the same amount of 0.002 *N* potassium iodate solution (Section 12 (*g*)) to the other sample. Both samples must now show a blue color and contain the same amount of added 0.002 *N* potassium iodate solution. Titrate the solution in dish A with 0.002 *N* thiosulfate solution (Section 12(*f*)) to the disappearance of the blue color and record the number of milliliters required as T_{A}. Similarly titrate the solution in dish B and record as T_{B}.

(*d*) *Titrating* (*Potentiometric Titration*).—Employ the apparatus of Section 5(*c*), (*d*), (*f*), (*g*), (*h*), and (*j*), and the general procedure of Section 7(*c*). Empty bottle A into the clean, 800-ml beaker, rinsing the bottle with a few milliliters of distilled water and adding the rinsings to the beaker. Rinse both electrodes with distilled water and readjust the sleeve on the calomel electrode to provide a fresh junction. Place the beaker on the titrating stand with both electrodes immersed in the sample, start the stirrer, and adjust its speed to mix the sample rapidly without causing a vortex to draw bubbles of air into the liquid. Add about 3 ml of starch solution (Section 12(*h*)) to the sample. If a blue color appears, add about 1 ml, measured to the nearest 0.01 ml, of 0.002 *N* potassium iodate solution (Section 12(*g*)) to the sample. If the sample remains colorless, add sufficient 0.002 *N* potassium iodate solution to develop a blue color and then 1 ml in excess, measuring the total addition to the nearest 0.01 ml. Titrate with 0.002 *N* sodium thiosulfate (Section 12(*f*)). Record the cumulative amount of titrant, the emf across the electrodes, and the quotient

$$\frac{\Delta\,\text{emf}}{\phantom{\Delta\,\text{emf}}}$$

thiosulfate after each addition. Make progressively smaller additions of thiosulfate as the end point is approached and passed. Record the cumulative addition corresponding to the maximum value of the quotient, without regard to sign, as T_{A}. Empty the beaker, rinse it and the electrodes with distilled water, and prepare a fresh junction with the sleeve of the calomel electrode. Empty bottles B and C into the beaker, rinsing bottle B with distilled water. Add 3 ml of starch solution and the same amount of 0.002 *N* potassium iodate, measured to the nearest 0.01 ml, as was added to the sample from bottle A. Titrate this sample exactly as before and record the result as T_{B}.

(*e*) *Titrating* (*Dead-stop End Point*)—*Adjustment of Apparatus.* Turn on the apparatus 10 to 15 min before a titration is to be made. Add 500 ml of distilled water to the beaker (Section 5(*c*)). Start the stirrer. Using the general technique of Section 13(*b*), add 3 ml hydrochloric acid (Section 12(*d*)) to the beaker, then 1 ml of potassium iodide solution (Section 12(*a*)), and finally 1 ml of manganous sulfate solution (Section 12(*b*)). Then by means of the buret add 0.5 ml of 0.002 *N* sodium thiosulfate (Section 12(*f*)). With the "coarse" and "fine" adjustments, set the "eye" (see note 20) so it is approximately 80 per cent open. The apparatus is now ready for the titration.

NOTE 20.—This method is based on polarization and depolarization of plain platinum electrodes immersed in the solution to be titrated. The condition of the electrodes and therefore the end point is determined by the appearance of an indicator ray tube of the 6E5 type (also referred to as a "magic eye"). When the solution is "reducing," as when thiosulfate is in excess, the electrodes become polarized ("eye" opens); when the solution is oxidizing, as when iodate is in excess, the electrodes become depolarized ("eye" closes). The end point is much more sensitive when the titration is carried out with iodate. Consequently an excess of thiosulfate is added.

NOTE 21.—By adding the fixing reagents in reverse order, a solution of the same general properties as that obtained in the titration is obtained. By adding the excess sodium thiosulfate solution, the solution is made "reducing," thus making possible a setting of the "eye." If the apparatus is used regularly, such adjustment generally is not necessary.

(*f*) *Actual Titration.*—Using the same general technique of Section 13(*c*), pour all of the solution from bottle A into the beaker. With the stirrer running, add 0.50 ml of 0.002 *N* thiosulfate by means of the buret. This should cause the "eye" to open to about 80 per cent of full opening area. Use the fine adjustment if required to set the "eye." Then add 0.002 *N* potassium iodate dropwise until 1 drop causes the "eye" to close. This is the end point. Subtract from 0.50 the milliliters of 0.002 *N* potassium iodate required and record this value as T_A. Empty the beaker. Empty bottles B and C into the beakers, rinsing with distilled water. Add 0.50 ml of 0.002 *N* sodium thiosulfate. Then add 0.002 *N* potassium iodate dropwise until one drop causes the "eye" to close. Subtract from 0.50 the milliliters of 0.002 *N* potassium iodate required and record the result as T_B.

NOTE 22.—It should be noted that the 0.50 ml of 0.002 *N* thiosulfate added is equivalent to 0.032 ppm of dissolved oxygen. If more than 0.032 ppm of dissolved oxygen is present, the "eye" will not open and more thiosulfate must be added until the "eye" opens. In calculating T_A and T_B any additional thiosulfate added must be considered.

Calculation

14. Calculate the dissolved oxygen content of the sample as follows:

$$\text{Dissolved Oxygen, ppm} = 0.064\,(T_A - T_B)$$

Precision and Accuracy

15. A skilled operator employing this method can obtain results with a precision of ... ppm and an accuracy of ... ppm.

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DISCUSSION

MR. ROBERT C. ADAMS*[1] (*presented in written form.*)—Frequently compliments to the author of a paper are empty words from a similar source. In this case, however, I believe that the Society owes a particular debt to the author which should be acknowledged.

Mr. Riedel is not a member of the Society, Committee D-19, or the Task Group which planned this study. But he was the man who made all preliminary arrangements at the site, received and sorted the equipment supplied by the cooperators, and supervised the entire group in carrying through the planned program. Now he has prepared a comprehensive report of the tests, the data, and the significance of those data. Let me express our appreciation to Mr. Riedel for his large part in this cooperative effort.

This report is another example of the distressing fact, so well known by technologists, that an investigation seldom produces unequivocal answers to the questions studied. A corollary is that too often a study poses more questions than it answers.

Basically, this program was planned to answer three questions:

1. Is Method I the best available?
2. Is Method III almost as good?
3. Is Method VI good enough to keep as an alternate ASTM procedure?

Desire for quantitative answers dictated the careful statistical planning, supervision, and subsequent analysis. Many variables were introduced and considered to provide more comprehensive information. But the basic questions were the three above, and how well have they been answered?

This paper gives affirmative answers to all three questions. The standard deviation of Method I was consistently lower than that of the others. That of Method III was higher, but not significantly so, so that Method III can be accepted as virtually equivalent to Method I. Method VI was distinctly less precise, yielded consistently high results, and could not cope with interference. Despite these shortcomings, it is useful in limited applications, particularly where a simple and rapid procedure must be employed. These general replies are somewhat less than the hoped-for quantitative ones.

This is at least the third attempt to obtain a definitive answer to the first question. The first was by an earlier task group of Committee D-19 in a somewhat similar clinic in 1944; the second, that of the Heat Exchange Institute. All agree on the superiority of Method I, but cannot convincingly say how good it is. This paper says that at least two thirds of successive determinations from a uniform source will fall within a range of 0.005 ppm. The unpublished data of the 1944 clinic indicated a corresponding range of about 0.004 ppm. A comparable figure is not available from the HEI study because of the different statistical collection and analysis of data used therein.

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* Opinions expressed herein are those of the discussor and not necessarily those of the Department of the Navy.

[1] Superintendent, Chemical Engineering Laboratory, U.S. Naval Engineering Experiment Station, Annapolis, Md.

These independent and consistent judgments of Method I as the best are gratifying to me. Method I is the one developed by my associates and described to the Society in our 1943 paper.[2] Its major use by us has been for qualification testing of deaerating feedwater heaters. In such applications, of course with skilled and experienced analysts, we have felt that the range as indicated above (2 σ) was no more than 0.002 ppm. This is an opinion, not a calculated value.

While this report returns another vote of confidence in the superior precision of Method I, it rouses suspicions of the accuracy of this and all the other methods. I refer to the data in Table II of the paper. As the author points out, the results obtained on concentration C, that containing reducing interference, are lower by all methods than can be rationalized. Our 1943 paper presented an exploration of all types of potential interference and concluded that Method I should compensate for any whose chemical behavior was not identical with that of dissolved oxygen. After the years of use and study of this method by ourselves and others, this questioning of compensation for reducing interference is most disturbing. Here, the study intended to answer questions asks a more serious one, which must be answered.

Let me urge that studies of relative accuracy, as well as precision, be planned and prosecuted as soon as possible. I suggest that Methods I, III, and VI of this paper be used for such further comparative study.

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[2] Robert C. Adams, Robert E. Barnett, and Daniel E. Keller, Jr., "Field and Laboratory Determination of Dissolved Oxygen," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 1240 (1943).

PROPERTIES OF VARIOUS FILTERING MEDIA FOR ATMOSPHERIC DUST SAMPLING*

BY WALTER J. SMITH[1] AND NORMAN F. SURPRENANT[1]

In sampling for atmospheric dust and for testing atmospheric dust conditions, a number of methods are in use which depend upon filtration to arrest the dust particles. The effectiveness of any such method or even its success can depend, to an important degree, on the filter medium that is selected. Because they may be so important, the properties of any filter medium should be well understood before its use is recommended for any test method. It is the purpose of this paper to compare and discuss properties of several filtering media with respect to various air sampling requirements. All of the media to be considered are now available, and most of them are being used for air assay work.

There are various reasons for collecting a sample of atmospheric dust, and the purpose to be served will influence selection of the filtering medium. Measurement of mass concentration of dust in the air, particle size distribution, chemical analysis of the particulates, toxicity assay, radioactivity measurements, study of organisms, and evaluation of soiling characteristics are some of the reasons for or purposes of sampling.

Conditions under which the sampling must be done may also influence selection of a medium. For example, glass fibers would not be used in an atmosphere known to contain an appreciable amount of hydrofluoric acid vapor.

In some cases a particular filter medium is used in a certain application only because of long standing practice which, for consistency, is kept unchanged. However, when the need arises to select a filter for some new or special purpose, an understanding of the general filtering properties of available media should be useful in making an intelligent choice. It is our purpose to contribute to the fund of such information.

The problems associated with selection and use of air sampling filter media were discussed at the Air Cleaning Seminar, sponsored by the Atomic Energy Commission and held at Ames, Iowa, September 14 to 17, 1952. As a result of that meeting, a study of filter media and sampling practices was undertaken by Arthur D. Little, Inc. A questionnaire survey of some 40 laboratories, most of them within the Atomic Energy Commission operating areas but including also a number of outside laboratories, provided a list of air sampling media that are in current use at these laboratories.

A group of samples was then assembled that represented nearly all of the media mentioned in the survey. This paper describes and discusses air filtration test results obtained for these media and for a few others[2] that were included because of their special interest. The test methods have included di-octyl phthalate smoke penetration, atmospheric dust penetration, efficiency by particle size, and plugging rate on atmospheric dust. A range of performance characteristics is provided which may aid one in selecting

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28-July 3, 1953.

[1] Arthur D. Little, Inc., Cambridge, Mass.

[2] Atomic Energy Commission mineral papers were added to the group.

TABLE I.—EFFECT OF FLOW RATE ON PRESSURE DROP[a] AND DOP SMOKE PENETRATION[b] FOR VARIOUS AIR SAMPLING MEDIA.

Flow Rate, linear ft per min		AEC No. 1	CWS No. 6	HV 70		Hurlbut Glass Paper	Whatman Chemical Filter Papers									S & S No. 604	Membrane Filters		AEC Mineral Filters		MSA Type "S"
				9 mil	18 mil		No. 1	No. 4	No. 32	No. 40	No. 41	No. 41H	No. 42	No. 44	No. 50		"HA"	"AA"	Glass-Asbestos	All-Glass	
5	Penetration	0.022	0.015	5.0	0.47	0.001	73.0	84.0	3.7	32.0	89.0	93.0	5.0	6.5	30.0	93.00	0.002	0.002	0.029	0.008	45.0
	Pressure drop	0.7	0.67	1.1	1.2	1.05	1.9	0.5	7.2	2.5	0.35	0.45	8.7	7.7	9.5	0.35	5.4	2.2	0.7	0.75	0.3
10	Penetration	0.036	0.023	5.0	0.53	0.001	68.0	81.0	3.0	26.0	84.0	89.0	2.0	4.5	13.0	90.0	0.002	0.002	0.052	0.020	50.0
	Pressure drop	1.45	1.45	2.2	2.45	2.2	3.75	0.95	14.6	5.1	0.68	0.9	17.0	15.3	17.7	0.7	10.9	4.3	1.45	1.45	0.6
20	Penetration	0.045	0.04	3.5	0.65	0.003	43.0	77.0	0.45	16.0	77.0	81.0	0.75	1.4	3.0	85.0	0.01	0.002	0.071	0.038	52.0
	Pressure drop	2.9	2.9	4.6	4.9	4.4	7.7	1.95	27.3	10.7	1.35	1.9	33.0	28.6	35.2	1.45	21.6	8.5	3.0	3.05	1.05
28	Penetration	0.055	0.057	2.0	0.69	0.005	27.0	73.0	0.35	8.0	75.0	76.0	0.22	0.5	0.9	79.0	0.015	0.01	0.073	0.05	52.0
	Pressure drop	4.2	4.05	6.3	6.9	6.1	10.6	2.8	38.0	15.0	2.0	2.7	45.5	40.0	48.5	2.1	31.0	11.8	4.25	4.25	1.6
50	Penetration	0.045	0.045	1.7	0.45	0.005	11.0	62.0	0.3	2.8	67.0	65.0	0.02	0.2	0.15	67.0	0.015	0.015	0.08	0.051	51.0
	Pressure drop	6.7	7.5	9.4	13.0	10.8	19.6	5.5	69.4	25.3	3.8	5.5	81.0	71.0	86.0	3.9	59.6	24.5	7.8	7.7	3.0
100	Penetration	0.031	0.037	0.2	0.1	0.005	1.2	25.0	...	0.23	44.0	34.0	...	...	...	39.0	...	0.02	0.04	0.025	45.0
	Pressure drop	13.3	17.0	21.8	27.0	19.8	40.5	11.5	...	54.0	8.1	11.5	...	...	...	8.5	...	39.0	16.0	15.2	6.5
150	Penetration	0.021	0.018	0.1	0.025	0.003	0.3	12.0	...	...	29.0	21.0	...	...	...	18.0	...	...	0.018	0.013	34.0
	Pressure drop	22.5	25.5	34.5	38.2	32.5	60.0	18.1	...	...	12.5	17.0	...	...	...	15.0	...	...	26.7	25.0	10.8
200	Penetration	0.011	0.01	...	...	...	...	...	...	...	15.0	13.0	...	...	...	7.0	...	...	...	...	28.0
	Pressure drop	29.5	35.0	...	...	...	...	...	...	...	17.8	24.0	...	...	...	22.7	...	...	...	...	16.3

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[a] Pressure drop in inches of water.

[b] DOP smoke penetration in per cent (di-octyl phthalate particles 0.3 μ diameter, 50 micrograms per liter of air).

a filter material for any dust sampling purpose.

DI-OCTYL PHTHALATE SMOKE PENETRATION TEST

The di-octyl phthalate smoke penetration meter, or "DOP tester" as it is called more commonly, was developed by the armed services during the war and has become a well known and highly respected device for evaluating high efficiency filters. Instrumental parts of the tester and theories of their operation have been well presented in the literature (**1, 2, 3**).[3] For our needs here a very brief description will serve. In the DOP tester, there is a smoke generator for producing a controlled, mono-dispersed, liquid aerosol of di-octyl phthalate. This is accomplished by condensation from the vapor state, and the droplets so formed are held very close to 0.3 μ diameter. Particle loading is about 50 micrograms per cu dm. A light scattering chamber is also provided with sensitive photoelectric pickup means for accurate measurement of smoke particle concentration. The tester is adjusted against full aerosol concentration (unfiltered smoke) and against absolutely clean air. Penetration through a test specimen of filter medium is then read off directly in per cent.

Since the aerosol particles at 0.3 μ diameter are in the approximate size range for the most numerous microscopically visible atmospheric dust particles, the DOP test gives efficiency values that parallel those obtained by atmospheric dust counts.

Under the somewhat standardized conditions of normal laboratory test procedure, DOP smoke penetration measurements are made at 28 linear ft per min through a 4.5-in. diameter circular area of the medium. In the work to be described, this area size was used for flow rates up to 28 ft per min. But to reach the higher flow velocities (up to 200 linear ft per min), a test area of 1.75-in. diameter was used.

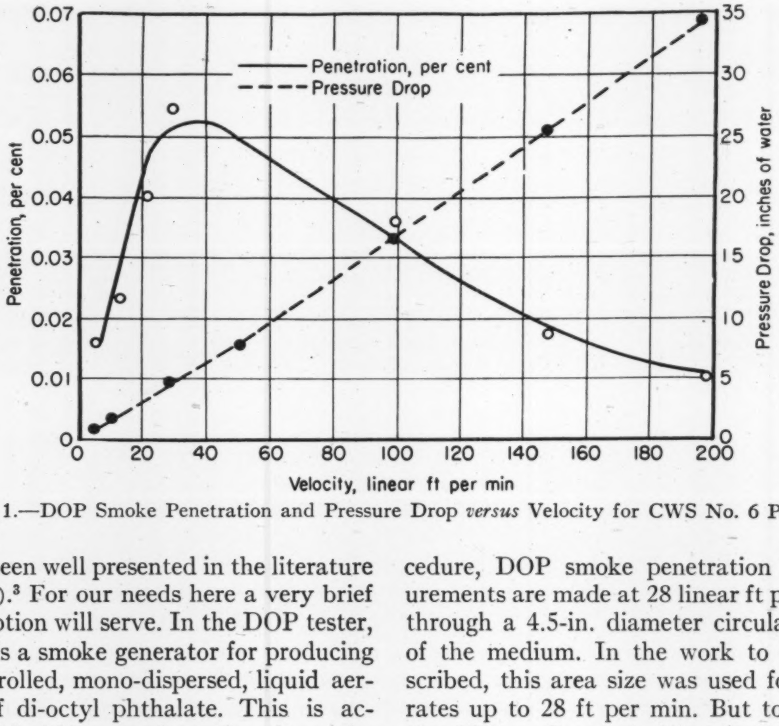

FIG. 1.—DOP Smoke Penetration and Pressure Drop *versus* Velocity for CWS No. 6 Paper.

Table I shows DOP smoke penetration efficiencies over a range of air flow velocities for the entire group of air sampling media. It is evident immediately that there is a very great difference in efficiencies as measured by this test. Perhaps this is the point at which to stress that DOP smoke penetration alone must not be taken as a general measure of usefulness for all filters. It is a very severe test and is now used primarily to rate absolute-type filters. When dealing with media intended to collect bulk dust or to analyze for atmospheric dust on a weight basis, very fine particles contribute to a minor degree and become unimportant; the DOP test then has much less significance. However, if one's interest extends to the submicron size dust particles of the atmosphere (and these are by far the most numerous), then the DOP test can tell a great deal about what a filtering material can be expected to do.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1133.

An interesting feature of the data shown in Table I is the relation of DOP filtering efficiency to flow velocity for the different types of filter materials. Sets of data selected from Table I have been plotted to show some characteristic curves.

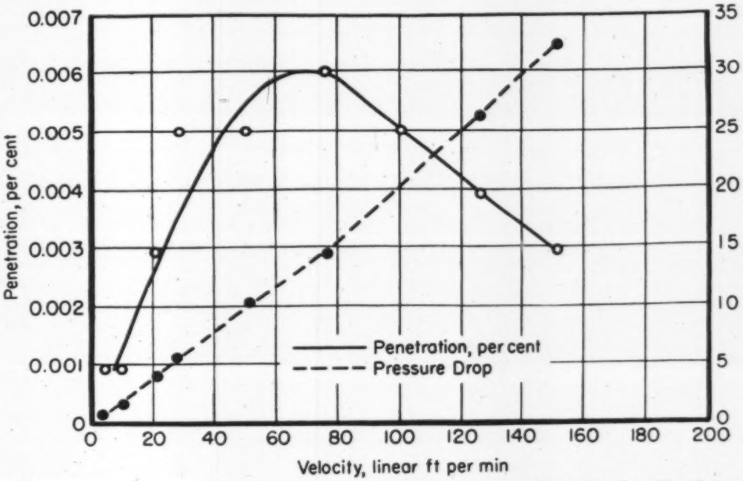

FIG. 2.—DOP Smoke Penetration and Pressure Drop *versus* Velocity for Hurlbut Glass Fiber Paper.

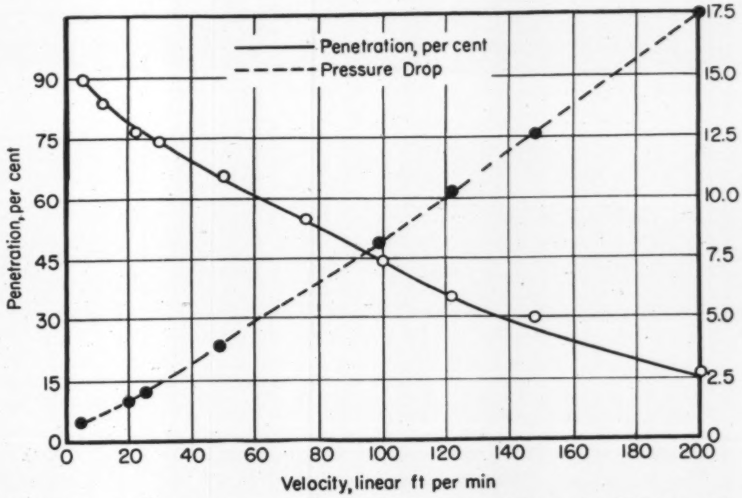

FIG. 3.—DOP Smoke Penetration and Pressure Drop *versus* Velocity for Whatman No. 41 Paper.

Figure 1 is for CWS No. 6 paper. At

a low air flow rate, it is very efficient. This is a fortunate circumstance because this type of material is used principally for making large volume, high-efficiency space filters in which face velocity through the medium is only 5 linear ft per min. With increase of flow rate, smoke penetration increases to a maximum at about 30 ft per min. As the flow rate is further increased, penetration again falls off progressively. This behavior has been studied by Ramskill and Anderson of the Naval Research Laboratories (4). They attribute the low-velocity positive slope to the influence of diffusional collection, while the higher velocity negative slope is explained by influence of inertial effects. In addition to flow velocity, these authors show how the character of the curves is controlled by aerosol particle size, particle density, diameter of the filter fiber, and inter-fiber spacing.

Pressure drop, plotted separately in Fig. 1, is nearly linear with flow rate, indicating viscous flow through the medium.

All of the high-efficiency papers, AEC No. 1, AEC mineral fiber papers, and HV 70 (18, mil) show curves similar to that for CWS No. 6.

Figure 2 shows the plotted data for a still more highly efficient medium. This is a sample of glass fiber paper that contains a resin binder and is made by the Hurlbut Paper Co. The fibers in the sheet have a diameter of about $\frac{1}{2}$ μ. The resulting curve also shows the peak typical of high efficiency media.

Chemical filter papers as illustrated by the Whatman papers are made in several types, and they give a variety of curves. Figure 3 shows a plot for paper No. 41 which is typical of many of the cellulose papers.

Paper No. 42 (Table I) is remarkably efficient for an all-cellulose sheet. This efficiency is attained at low flow velocity, but pressure drop is high.

MSA type "S" filter, which is used successfully for high volume air sampling (5), shows an unusually uniform DOP efficiency level over a wide range of flow rates (Fig. 4). While all of the other filter specimens come in flat sheets, type "S" is different. It has a molded shape of concentric convolutions designed to provide a large filtering area. A piece was cut

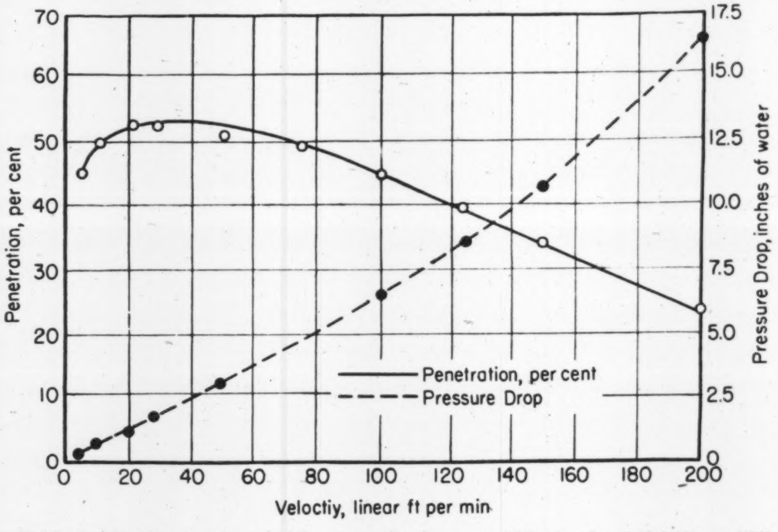

FIG. 4.—DOP Smoke Penetration and Pressure Drop *versus* Velocity for MSA Type "S" Filter.

from a reasonably flat area and used as the test specimen.

Membrane filters have been described as molecular sieves. Collection appears to be almost entirely at the surface. It is perhaps for this reason that they fill up rapidly on an oil smoke (like DOP) and so may not show up to best advantage in this test.

TABLE II.—IMPACTOR COUNT EFFICIENCY ON SUBMICRON ATMOSPHERIC DUST PARTICLES COMPARED WITH DOP EFFICIENCY FOR VARIOUS AIR SAMPLING MEDIA.

Flow rate, 20 linear ft per min.

Filter Medium	Atmospheric Dust Count Efficiency, per cent[a]	DOP Efficiency, per cent[b]
Whatman Chemical Filter paper:		
No. 1	50.0	57.0
No. 4	15.0	23.0
No. 32	99.1	99.5
No. 40	85.1	84.0
No. 41	26.5	23.0
No. 41H	24.0	19.0
No. 42	98.8	99.2
No. 44	97.0	98.6
No. 50	92.0	97.0
No. 540	67.0	65.0
S & S No. 604	13.0	15.0
HV 70 9 mil	96.5	96.5
HV 70 18 mil	99.5	99.3
MSA type "S"	46.0	48.0
Millipore type "HA"	No particles found after 6 hr running	99.9+
Millipore type "AA"		99.9+
S & S ultra filter		...
Hurlbut glass paper		99.99+
		99.9+
CWS No. 6		99.9+
AEC No. 1		99.9+
AEC glass-asbestos		99.9+
AEC all-glass		99.9+

[a] Average of four tests.
[b] Calculated from Table I.

All fiber filter materials "fatigue" in the DOP tester. After running on DOP for several minutes, the smoke penetration increases. One explanation offered is that electrostatic effects in the filter body are lost due to accumulation of liquid. It is known that filters depending on electrostatic effects fail quickly when used on oily smokes, so there is some basis for the suggested explanation. For the present, it is only important to mention that a DOP test should be made over a short period of time.

EFFICIENCY BY ATMOSPHERIC DUST COUNTS

It was stated earlier that DOP test results are comparable with efficiency as measured by counts on atmospheric dust particles. This is shown by the data in Table II. Here the DOP filtering efficiencies of the various media are given, calculated from Table I, and atmospheric dust count efficiencies are shown for comparison.

To measure these efficiencies on atmospheric dust, a high-speed impactor (6, 9) was used for collection. Particle concentrations were measured before and after the filter. In most cases, four tests were made on each filter and 200 counts were made each time. Efficiencies were calculated from counts on the sonic velocity stage of the impactor; particles were 1 μ and smaller in diameter. No counts were obtained on the clean side of the very efficient media even after running the impactor for 6 hr. It should be borne in mind that the great numbers of atmospheric dust particles are less than 1 μ in diameter. Rating of a filter by counts on such dust is the same as rating that filter for performance in those small particles.

TABLE III.—PARTICLE SIZE DISTRIBUTION IN ATMOSPHERIC DUST BEFORE AND AFTER FILTRATION THROUGH DIFFERENT MEDIA.

Particle Diameter, μ	Count Analysis of Air-Borne Particles, per cent of each size					
	Unfiltered Air	Filtered Air				
		Whatman No. 1	Whatman No. 4	Whatman No. 41	Whatman No. 42	MSA "S"
Below 0.4	31.8	52.8	45.2	47.4	51.0	50.0
0.4–0.6	42.6	35.3	38.1	40.3	39.3	41.8
0.6–0.8	16.2	10.6	14.2	9.1	8.5	7.0
0.8–1.0	6.8	1.1	1.9	2.4	1.1	1.0
1.0–2.0	1.6	0.2	0.6	0.8	0.1	0.2
Over 2.0	1.0	...	...	...	...	...

NOTES.—Flow Rate: 20 linear ft per min through the medium.
Each value based on counts for two filters with no fewer than 40 counts each point each filter.
Particles collected sonic velocity impactor.
Counts and measurements by oil immersion microscope 1350 ×.

Even those who have been aware of

the relation of DOP efficiency to particle count efficiency may be surprised by the close correlation of these separate methods. The results strongly indicate that the DOP tester can be relied upon to evaluate all filter media with respect to efficiency against submicron size atmospheric dust particles.

EFFICIENCY BY PARTICLE SIZE

In the methods just described, only submicron size particles were dealt with. When larger particles are considered, attention becomes limited to the cellulose fiber filters on the list. Larger particles do not penetrate the other media of the group.

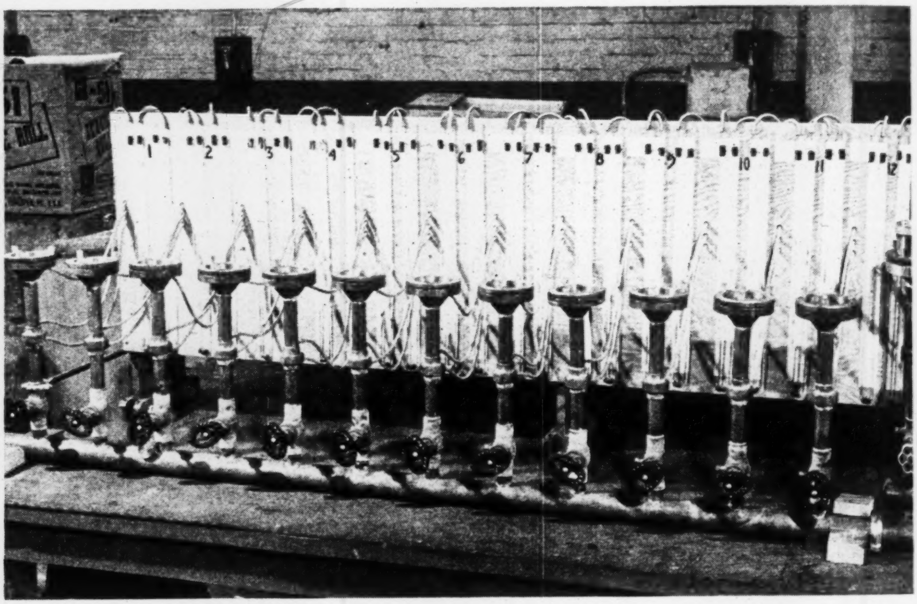

FIG. 5.—Life Testers for Air Filter Media.

Table III shows the particle size analysis for unfiltered laboratory air and for the same air after passing through each of several cellulose fiber filters. In every case, the count peak is shifted in the direction of the smaller particles, as would be expected. No particles larger than 2 μ were observed to have passed any of the filters. Time did not permit all of the cellulose fiber filters to be included, but a representative group was selected.

Efficiency of filtration by particle size is shown in Table IV. Here again efficiency was measured by particle count on high-speed impactor plates. No particles were found above the size of 2 μ, and all of the filters showed good to excellent efficiency on particles in the 1- to 2-μ range. When the primary interest is in weight of dust collected, these filters are generally adequate since large dust

TABLE IV.—FILTERING EFFICIENCY[a] BY PARTICLE SIZE FOR EACH OF SEVERAL AIR SAMPLING MEDIA.

Flow rate, 20 linear ft per min.

Particle Diameter, μ	Whatman No. 1	Whatman No. 4	Whatman No. 41	Whatman No. 42	MSA "S"
Below 0.4	57[b]	23[b]	23[b]	99[b]	48[b]
0.4–0.6	59	24	31	97	47
0.6–0.8	67	25	59	99	77
0.8–1.0	92	77	74	99	92
1.0–2.0	94	69	63	99	93
Above 2.0	100	100	100	100	100

[a] Efficiency for particle retention in per cent by count. Particles above 0.4 μ diameter collected by high-speed cascade impactor.

[b] DOP smoke value used for particles below 0.4 μ diameter.

particles contribute most. The weight contribution of a particle is measured by the cube of its diameter.

All of the results reported have been on fresh samples of media. Allowance should be made for the fact that all filters improve in efficiency as they fill. As a practical matter, all of the media tested here will perform much better in use than the figures indicate.

TABLE V.—LIFE TESTS—CHANGE OF PRESSURE DROP ACROSS AIR SAMPLING MEDIA WITH OPERATING TIME.

Figures show pressure drop in inches of water

Running Time, hr	AEC No. 1	CWS No. 6	HV 70		Hurlbut Glass Paper †	Whatman Chemical Filter Papers									S & S No. 604	Membrane Filters		AEC Mineral Filters		MSA Type "S"	Approximate Dust Load
			9 mil	18 mil		No. 1	No. 4	No. 32	No. 40	No. 41	No. 41H	No. 42	No. 44	No. 50		"HA"	"AA"	Glass-Asbestos	All-Glass		
FLOW RATE, 5 LINEAR FT PER MIN																					
0	0.72	0.72	0.95	1.05	0.95	1.9	0.48	7.0	2.45	0.35	0.5	8.5	8.0	9.5	0.35	5.4	2.3	0.7	0.7	0.17	63 micrograms per cubic meter
24	0.75	0.75	1.15	1.2	1.0	5.2	0.80	9.45	5.7	1.2	0.85	28.0	16.5	27.0	0.45	5.45	2.5	0.75	0.7	0.17	
48	0.75	0.75	1.2	1.25	1.0	6.2	1.15	11.0	6.85	1.5	1.65	...	...	...	0.8	5.5	2.6	0.75	0.7	0.2	
120	0.85	0.8	1.4	1.4	1.1	7.3	2.3	12.8	8.2	2.15	3.3	...	...	...	1.8	5.7	2.6	0.8	0.7	0.2	
192	0.85	0.9	1.55	1.7	1.1	8.0	3.2	...	9.0	2.7	4.5	...	...	...	2.6	5.9	2.8	0.9	0.75	0.22	
288	0.95	1.0	1.8	1.95	1.2	8.5	3.9	...	...	3.35	6.8	...	...	...	3.15	6.4	3.0	1.0	0.85	0.25	
336	1.00	1.05	1.9	2.0	1.2	...	4.15	...	...	3.9	...	...	...	...	3.45	6.7	3.1	1.2	0.85	0.25	
384	1.05	1.05	1.9	2.0	1.25	...	4.15	...	...	4.0	...	...	...	...	3.5	7.1	3.15	1.25	0.85	0.25	
480	1.05	1.1	1.95	2.1	1.25	...	4.25	...	...	4.3	...	...	...	...	3.55	8.1	3.2	1.35	0.85	0.25	
FLOW RATE, 28 LINEAR FT PER MIN																		FLOW RATE, 28 LINEAR FT PER MIN			
0	5.6	6.15	11.0	11.7	7.0											8.1	3.2	7.6	4.75	1.2	
24	5.8	6.35	11.5	12.1	7.15											8.2	3.15	7.8	4.75	1.2	
48	6.1	6.6	12.6	14.2	7.4											8.5	3.2	8.3	5.0	1.2	
72	6.6	6.85	...	...	7.6											8.55	3.3	...	5.25	1.25	
120	7.0	7.4	...	...	8.4											8.6	3.4	...	5.6	1.35	

LIFE TESTS

In many air sampling applications, plugging rate of a filter medium is not a problem. But in those cases where it is desired to sample over a long period of time or to accumulate a sizeable quantity of particulate matter, the rate at which plugging occurs may become important. At times flow resistance or the development of flow resistance may even determine the feasibility of taking the sample.

A life test or plugging rate test consists of operating a filter sample at some selected flow rate and observing the increase in pressure drop with time. The kind of equipment that was used for this is shown in Fig. 5. It consists of separate test stations in which samples of filter materials may be mounted and operated over long periods of time. Each station has a sample holder that takes a $3\frac{1}{2}$-in. diameter disk of the medium and exposes a test area 3 in. in diameter. A calibrated orifice meter and control valve allows each sample to be run at a selected rate. The testers were arranged in two banks of twelve units each; all twelve stations in a bank flowed into a single manifold line that was connected to the intake port of a three-stage Spencer turbine blower.

The flow rate tends to fall off, of course, as the filter fills with its accumulated dust load. This necessitates periodic adjustment of the valve to restore the proper rate. Pressure drop across each test specimen is measured with a U-tube water manometer.

TABLE VI.—SOME GENERAL PROPERTIES OF AIR SAMPLING MEDIA.

Material	Type or No.	Manufacturer or Source	Thickness, in.	Ash Content, g per 9 cm diam circle (unless otherwise stated)	Description	Present Application in Air Sampling
Chemical filter papers	Whatman 1	W. & R. Balston Ltd., England	0.008	0.00039	White cellulose papers of various grades	Tests depending on discoloration or change in light transmission.
	4		0.008	0.0005		
	32		0.010	0.00022		
	40		0.010	0.00009		
	41		0.010	0.00009		
	41H		0.007	0.00004	Nos. 41H, 50, and 540 are hardened papers	Analysis of particulate by destruction of medium.
	42		0.010	0.000064		
	44		0.008	0.000051		
	50		0.005	0.00016		
	540		0.006	Low ash		
	S & S 604	Schleicher & Schuell Co.	0.008	0.0002	...	...
Membrane filters	"HA"	Lovell Chemical Co.	0.005	1.5 per cent	Porous cellulose ester films	Particulate counts, identification by microscope. "Final stage" for impactor α counting.
	"AA"		0.005	1.5 per cent		
	Ultra Filter membrane type	S & S Co.	0.005	...		
HV 70	9 mil	Hollingsworth & Vose	0.009	14 per cent	Asbestos bearing cellulose base paper	General air assay and radioactivity monitoring.
	18 mil		0.018	14 per cent		
MSA	"S"	Mine Safety Appliances Co.	0.040	1.3 per cent	Molded cellulose—concentric convolutions	High volume air sampling.
Glass paper	...	Hurlbut Paper Co.	0.010	95 per cent	Fine glass paper—resin binder	High efficiency particulate removal.
CWS	6	Hollingsworth & Vose	0.030	11 per cent	Felt-like paper asbestos and cellulose	High efficiency particulate removal.
AEC	1		0.030	13 per cent		
AEC mineral filter	Asbestos glass	Arthur D. Little, Inc.	0.030	95.0 per cent	Glass and asbestos	High efficiency particulate removal.
			0.030	95.0 per cent	All glass	
					Both with resin binders	

It seems best to test all of the media at the same time so that there will be no question of varying dust conditions in the air. This brings up the matter of flow rate at which to run; for direct comparisons, all should be run at the same rate. The very low rate of 5 linear ft per min was selected as a start with the intention of increasing the rate after the rapidly plugging samples had been removed. When pressure drop became too high for any manometer, that test was stopped. After 480 hr of running, the flow rate was stepped up to 28 linear ft per min for all surviving specimens except the membrane filters. Only seven specimen filters were remaining 120 hr later. Atmospheric dust loading over the time period of the run was measured by weighing the total dust load on a membrane filter.

Table V includes life tests for the entire group of specimens. With one exception, the test specimens were flat disks. The exception, MSA type "S," as mentioned before is a molded filter with concentric convolutions. A whole filter was used and air flow adjusted to allow for the greater area, estimated to be 75 sq in.

It is interesting that the media that plug most rapidly are not necessarily the most efficient nor those with highest initial pressure drop. As a class, the chemical filter papers tend to plug most readily. High-efficiency papers show much better life. The membrane filters are very interesting: pressure drop is high initially but increases only a little as dust load accumulates.

To the best of the authors' knowledge, the rate at which a filter becomes loaded does not determine its life; regardless of time the pressure drop through a sampling filter is fixed by the amount of accumulated dust. Operating at low flow rate merely extends the time; dust loading in the air (assuming a constant dust composition) and the total amount of air passed are the controlling variables. In the life tests conducted at Arthur D. Little, Inc., very low flow rates were used. For this reason Table V gives a slow motion picture of plugging rates. Life for any other flow or dust loading can be estimated from the data given.

DISCUSSION OF FILTER PROPERTIES

For convenience of reference, Table VI contains some general information on the various filter media that have been discussed. This table is not complete in any way. It contains some of the more obvious qualities along with a few measurements of our own. Values for ash content of the chemical papers were given by the manufacturers. Values for other media were determined at our laboratory. Very often some special property will determine the suitability of a given material. Such properties are important and must be considered along with filtering performance when a sampling medium is being selected.

Chemical filter papers appear to be used more widely than any other type of air assay medium. This may be because they are nearly always at hand in a laboratory. For those purposes where the filter must be destroyed to isolate or concentrate the dust, the low-ash chemical filter papers are particularly useful.

High-surface reflectance of light from chemical papers have made them popular for those test methods that are based on discoloration of the collecting surface.

Although chemical filter papers probably were never intended for air sampling work, they have proved to be most popular. Many kinds are available and data in the tables of this report show the range of performance characteristics that can be expected. There are some properties inherent in paper and other fibrous media that are disadvantageous in some cases. These are mentioned at the end of this discussion.

Chemical papers in particular often are found to contain pinholes. When this occurs, it is likely that even some very large dust particles will penetrate.

Unless an air filter medium is manufactured especially for the purpose, its performance characteristics are likely to vary from lot to lot. Chemical filter papers are manufactured for chemical laboratory work. They are made and used primarily for wet filtrations. Therefore it is not surprising that wide variation in air filtration is often found for them. Table VII lists some experimental results that illustrate this point.

TABLE VII.—VARIATIONS IN DOP SMOKE PENETRATION AND PRESSURE DROP AT 28 FT PER MIN FOR VARIOUS SAMPLES OF CHEMICAL FILTER PAPERS.

Whatman Filter Paper	Reported Results (Table I)		Range		Number of Boxes Tested[a]
	Pressure Drop, in. of water	Penetration, per cent	Pressure Drop, in. of water	Penetration, per cent	
No. 1	10.6	27.0	9.5–12.8	12–28	5
No. 4	2.8	73.0	2.2–2.8	72–75	2
No. 32	38.0	0.35	38–45	0.008–0.35	1
No. 40	15.0	8.0	13–15	8–13	2
No. 41	2.0	75.0	2.0–4.2	49–75	4
No. 41H	2.7	76.0	2.7	76–82	1
No. 42	45.5	0.22	44–55	0.05–0.9	4
No. 44	40.0	0.5	40–48	0.25–0.5	1
No. 50	48.5	0.9	48–61	0.3–1.2	2

[a] Three samples tested in each box.

The membrane filter is relatively new, but it holds great promise as an all-round assay medium (**7, 8**). It is highly efficient, may be obtained in white or black, and collects particles only on the surface; refractive index is such that the filter structure itself becomes invisible for oil immersion microscope viewing. The filter can be dissolved if need be or it may be destroyed in other ways. Because they are very delicate, the membranes must be handled carefully and supported during use. To generalize, there appear to be more useful properties associated with membrane filters than with any other one medium.

The felt-like papers CWS No. 6, AEC No. 1, and the AEC mineral fiber papers were designed for efficient air cleaning and serve that purpose effectively. They are not so well suited for most assay work. Dust particles penetrate the structure so that they are buried and lost for some types of radioactivity measurements (α counts). These papers are so high in ash that they are not at all usable where the filter must be destroyed to perform analysis of the dust. If suitable precautions are taken, they may be used for gravimetric sampling on even the finest of dusts and fumes.

HV 70 is a closely formed paper and has found use particularly in radioactivity monitoring.

All-glass papers, like those developed by Naval Research Laboratories (**10**) and made to a limited extent by several paper companies, are to be recommended for high temperatures or in the presence of corrosive fumes or gases. In the series used by our laboratory, the Hurlbut glass paper serves as an example. These papers are made of very fine glass fibers and are the most efficient of fibrous filters. Some have resin or other binders that should be burned out before using the sheet in most kinds of test work. In gravimetric work care must be taken that loose fibers are not lost from the sheet.

All fibrous filters, cellulose or glass, have water associated or adsorbed in their structures. The amount depends upon atmospheric humidity and will vary. In weighing the amount of dust load collected by such filters, it is very important to condition the filter at a known humidity level before every weighing and to weigh the filter in a closed container.

Dust collected on a fibrous filter will

penetrate the filter body to some extent. For this reason it is very difficult, if not impossible, to make dust studies under the microscope on most paper filters.

SUMMARY

A group of atmospheric dust sample media has been studied for performance characteristics. The media were selected to represent those in use in a number of laboratories. Test methods used were dioctyl phthalate smoke penetration, atmospheric dust penetration, efficiency by particle size, and plugging rate on atmospheric dust. A wide range of properties was shown.

The filtering properties have been discussed and the suitability of the media for various applications has been indicated.

It has been demonstrated that efficiency measurements by the DOP smoke test follow very closely the results given by atmospheric dust counts. This suggests that the fast DOP method can be used to rate any filter medium on per cent of atmospheric dust penetration by particle count.

Acknowledgment:

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DISCUSSION

Messrs. J. E. Yocom and I. M. Saslaw[1] (*by letter*).—We feel that this is an excellent piece of work and a valuable contribution to the standardization of air-pollution measurements, but we do have comments on specific points.

Particle collection efficiencies were based entirely on an impactor. We feel that additional discussion of the use of this method would be helpful. In addition, there is some question of an impactor's efficiency on particles under 1 μ, and there is always the possibility that the fracturing of larger particles can contribute to the number of smaller particles.

The paper points out that most of the media tested were very efficient in collecting atmospheric dust on a weight basis—that essentially all particles greater than 2 μ were collected. However, from Table III, showing the size analysis of typical atmospheric dust, it would appear that there are very few particles greater than 2 μ. It does not seem obvious that this small number of particles could contribute sufficient weight to show a high weight efficiency of collection and give a representative sample for chemical analysis.

It would be helpful to have shown in Table II the over-all calculated filtering efficiency of each of the media on a weight basis as a guide in chosing a medium for collection of a dust sample for quantitative analysis.

There is some question as to the relationship between Tables III and IV in the lowest two-size classes. With the data on particle-size distribution of unfiltered air in Table III and the percentage removed from each size range in Table IV, the particle-size distribution of dust in air filtered through each of the media shown can be calculated. These values should check exactly with the distribution shown in Table III. However, they do not check for the categories "below 0.4" and "0.4–0.6." They are close in agreement for "0.6–0.8," and they check very well for all ranges above. It is possible that some of the figures in categories 1 and 2 above have been transposed.

Mr. W. J. Smith (*author's closure*).—No detailed discussion was given on the impactor and its method of use because we felt that this information was well presented in the references indicated.

The sonic velocity impactor has been shown (1) to have excellent efficiency of collection down to 0.2 μ diameter. We realize that collection is not one hundred per cent. However, since the same impactor is used before and after the test filter medium, collection efficiency for a given size of particle automatically cancels out.

Large particles are caught on one or another of the lower speed stages in the cascade impactor. Some of them may shatter on the plates, but the fragments are held by the adhesive. In our experience large particles have never been a source of any trouble.

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[1] Battelle Memorial Institute, Columbus, Ohio.

We would welcome further thoughts on how to collect and count the few particles that get through a good air filter. As it is, we must often run an impactor for hours to collect enough particles for a count. Collection of scarce particles is a real problem and we need some new ideas.

It is true that atmospheric dust in a quiet area will contain few particles larger than 2 μ diameter. However, since weight collection of dust depends upon the cube of particle diameters, a few large particles quickly out-weigh the more numerous small particles. It is for this reason that we regard weight collection efficiency as an unsatisfactory way of rating efficiency filters. Very few chance large particles can make a tremendous difference and lead to very uncertain results.

Because of our preference for count efficiency, we did not attempt to list weight efficiency values. Weight efficiency can be calculated from particle count and size distribution data such as is given in Table III. The calculation will show a high efficiency by weight for all of the media.

It is agreed that if all of the information in Tables III and IV was based on identical samples and if particle counting by size were an exact operation, the two tables should check exactly. Neither supposition is true so that a discrepancy between the tables could exist and does.

The calculation of particle size distribution in filtered air based on data for unfiltered air and filtering efficiencies does not match reported particle size distribution. The differences are such as to suggest an accidental transposal. This would be a comfortable solution to the discrepancy, but our recheck has shown no such error.

The reason for the discrepancy between Tables III and IV is that filtering efficiency for particles below 0.4 μ was measured by DOP smoke (0.3 μ diameter) penetration (Table II); all other observations were by atmospheric particle count. It is very difficult to estimate particle size in the 0.4 μ range by microscope; DOP smoke penetration is simpler and more precise. However, the two sets of data were taken on separate sets of test samples. Chemical filter papers can vary in performance (Table VII) and the variation may help to explain the differences noted.

INSTRUMENTATION AND ANALYTICAL TECHNIQUES FOR THE CONTINUOUS DETERMINATION OF AIR CONTAMINANTS*†

By Morris Katz[1] and George D. Clayton[2]

Synopsis

This paper describes some instrumentation and analytical techniques which have proved useful in the study of air pollution with particular reference to the Windsor-Detroit area, under the terms of a joint reference submitted by the Governments of Canada and the United States to the International Joint Commission in 1949.

The methods of study of the atmosphere which are discussed include the determination of:

1. Aerosol contaminants by continuous paper filtration techniques followed by identification by chemical and spectrographic analysis;
2. Recording optical methods for light transmission in the atmosphere as affected by air pollution;
3. Gaseous and volatile contaminants such as sulfur dioxide, hydrogen sulfide, chlorides and fluorides, oxidant gases and ozone;
4. Meteorological factors such as wind direction, velocity, rainfall, lapse rates, and solar radiation.

The complex contaminants, released to the atmosphere by the varied domestic and industrial activities of man may be dispersed rapidly by mixing with cleaner air or more slowly to form a haze or smog. The great variability in the degree of contamination of the atmosphere is brought about by the interplay of meteorological and topographical factors. These cause the pollution load to vary with the time of day and season of the year. Continuous instrumentation and analytical techniques are therefore necessary if it is desired to obtain an adequate picture of the variation in intensity of gaseous and aerosol contaminants over a given city or industrial area. Intermittent samples collected over short periods of time may be useful in locating sources of pollution, but only a continuous technique will yield data truly representative of conditions which may form the basis of complaint.

Substances which pollute the atmosphere may be classified, simply, into (1) gases or vapor, (2) liquid droplets or solid particles under the general heading of particulate matter. The first group includes the true gases and compounds which have boiling points below about 200 C. The more important gaseous pollutants, derived principally from industrial sources, are sulfur dioxide, hydrogen fluoride, hydrogen sulfide, organic sulfides, and miscellaneous hydrocarbons which can undergo oxidation and polymerization to "smog gases." These smog gases have been the subject of considerable study in the Los Angeles area (**24, 30**),[3] but there is no reason to believe that they may not occur in other industrial areas. It would appear that ozone and oxides of nitrogen, either of natural or industrial origin, play a part in the conversion of hydrocarbon waste products to smog gases.

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

† Report No. 130, Defence Research Chemical Laboratories, Ottawa, Canada.

[1] Research Scientist, Defence Research Chemical Laboratories, Ottawa, Canada.

[2] Senior Sanitary Engineer, U. S. Public Health Service, Washington, D. C.

The particulate contamination is usually classified according to particle size into deposited matter (dustfall) and finely divided suspended matter (aerosols). The fly ash, coarse dust, and dirt which consist of particles larger than about 10 μ in diameter settle out of the air fairly rapidly and constitute the major portion of the deposited matter. The finer particles ranging from about 5 μ down to 0.1 μ or less form suspensions in air which are mechanically stable. The smaller particles in this range exhibit Brownian movement and may be dispersed in the atmosphere by wind currents almost like a gas.

The complex nature of atmospheric pollution in a number of industrial communities has recently been described by Cholak (**5**). He has discussed the range of variation in concentration of suspended matter, carbon dioxide, carbon monoxide, the halogens, sulfur compounds, ammonia, oxides of nitrogen, and a group of aldehydes. Katz (**19, 20**) and Clayton (**6**) have discussed the varied character of the gaseous and particulate pollution in the Detroit-Windsor area. Some of the unique aspects of the pollution problem in the Los Angeles area have been reviewed recently by Littman, Magill, and their associates (**24, 25**) and by Shepherd (**30**). It is evident from these investigations that the successful solution of the problem of instrumentation and analysis for the study of air pollution will require the use of all the newer techniques involving microanalysis, colorimetry, fluorometry, ultraviolet, visible and infrared spectroscopy, electrometric, and conductometric methods. The properties of aerosols may be influenced and altered by the method of collection; therefore it is important to develop more fully those optical methods capable of measuring properties *in situ*, such as light scattering. Mass spectrography has become an important tool in the identification of gases and vapors. The electron microscope and X-ray diffraction are proving of considerable value in identification of particulate matter. The various analytical methods which are likely to prove useful in a broad study of air pollution have been reviewed by Kingsley Kay (**22**), while Thomas has presented recently an excellent paper on the development of instrumentation for the continuous study of air pollution, including meteorological factors (**36**). Many of these developments have occurred within the past five years, as a stimulating aftermath of the air pollution disaster at Donora, Pa., in October, 1948.

PARTICULATE MATTER

Dustfall:

The determination of particulate matter on a continuous basis involves, in general methods, which distinguish between coarse particles that settle rapidly and fine suspended particles. The coarse particulate matter is collected in a variety of ways: on greased plates, trays, or petri dishes, or in dustfall cans or jars. Much useful information can be gained by dustfall jars distributed throughout the city area. In the Windsor region, dustfall cans at twenty stations have been employed to determine the total solids deposited in tons per square mile per month. The deposits have been analyzed chemically to determine the soluble and insoluble (in water) material, tar (soluble in CS_2), combustible matter, and ash. The dissolved impurities have been analyzed for sulfates, chlorides, and lime (19, 20). This survey has been used in conjunction with area sampling of suspended matter to delineate districts of high and low pollution. An important improvement in this type of collector is the recent development of a directional dustfall instrument by Munger of the Battelle Memorial Inst. This is described by Thomas (36) as operated by a wind vane controlled by the wind direction so as to move a slotted cover containing openings for eight glass jars, corresponding to eight points of the compass. A ninth jar is included for calm air conditions.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1156.

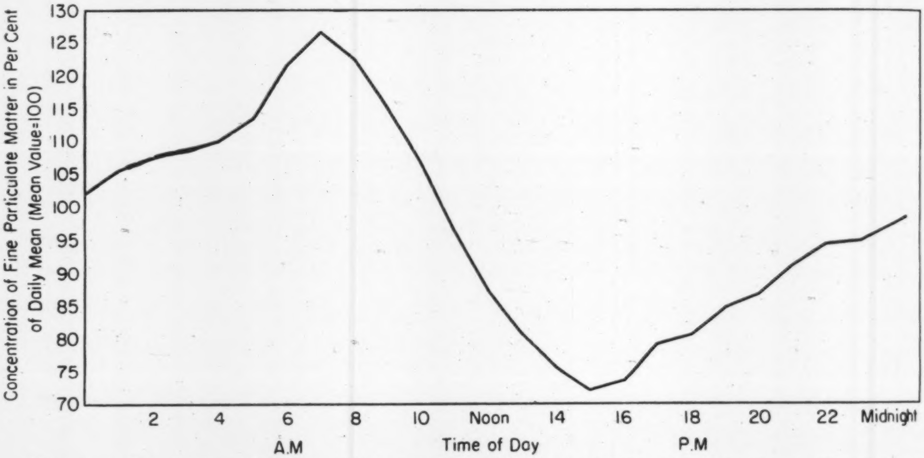

FIG. 1.—The Daily Variation of Suspended Particulate Matter in Downtown Windsor, as Determined by the Owens Automatic Paper Filtration Apparatus.

Suspended Particulate Matter:

Although coarse particles of fly ash and dusts constitute a nuisance in many cities and industrial areas by settling on lawns, parks, and buildings, the fine particles below 5 to 10 μ in size are usually considered to be of greater importance. Particles in the range of about 2 μ to less than 0.3 μ consisting of soot and tarry material, metallurgical and chemical dusts and fumes, are mainly responsible for soiling of textiles and walls, and form a haze which reduces visibility. They can be readily inhaled into the lungs and are therefore of considerable interest in any study of the effects of air contaminants on health.

The methods for the continuous instrumentation study of fine aerosols are mainly:

(*a*) The continuous collection by paper or membrane filtration methods,

(*b*) The measurement of the light scattering properties of fine particles, and

(*c*) The measurement of visibility.

Such instruments yield valuable information either directly or indirectly on the variation in intensity of fine particulate pollution, especially if supplemented by tests on the particle size distribution of the aerosols and chemical analysis of collected material.

Owens Recorder.—One of the oldest

instruments for the continuous measurement of suspended particulate matter was developed by Owens (27) in England many years ago. It has been used for the continuous determination of smoke concentrations in the air of a number of cities in Great Britain for over twenty years (8, 27). This instrument was used by Davidson (7) in a study of smoke conditions in New York City. More recently a modified form of this equipment has been used by Katz in the Windsor-Detroit study. A fairly complete description of the original instrument and the modifications made by Katz were presented by Thomas (35). The original instrument draws a measured volume of air (125 to 300 cu cm per min) through a small circular area ($\frac{1}{8}$ in. diameter) on the periphery of a filter paper disk. The paper is shifted to a new position every 15 min. In the modified form the sampling rate is 1 liter per min and the sampling time is 30 to 45 min. The spots are compared with standard shades of color in shade numbers from zero to 25.0. These shades are calibrated in terms of mass concentration in mg per cu m.

There is no doubt that useful information may be obtained by the Owens atmospheric pollution recorder. The diurnal variation of fine particulate pollution during the period February 26 to June 26, 1952, at Windsor, Ont., is illustrated in Fig. 1. The daily cycle shows a morning rise to a peak concentration at about 7.00 to 8.00 am and an afternoon minimum at about 3.00 to 4.00 pm. The concentration increases, thereafter, in a fairly regular manner throughout the night until the early morning maximum is attained. This cycle of particulate pollution follows the diurnal pattern of turbulence and temperature lapse rate in the Windsor area more closely than the sulfur dioxide pollution.

Autosampler.—An automatic filtration apparatus, designed by Hall (14) has been used recently in the study of aerosols in the Los Angeles area. This "autosampler" collects fine particulate matter by continuous filtration through Whatman No. 52 paper. A 25 cu ft volume of air is drawn at a uniform rate through a 1-sq in. area of filter paper during 1 hr. This cycle is repeated every hour on a fresh area of paper. At the same time a parallel air stream collects a 24-hr sample of the aerosols on an asbestos pad in a sufficient quantity to be weighable. The hourly spots or stains are evaluated by measurement of the reflectance in a Beckman DU spectrophotometer at a wavelength of 400 m μ. For comparison with similar samples of other volumes and areas, these readings are converted into a scale designated as K_m values—the mass equivalents of the deposit per cubic meter.

A model of the Hall instrument obtained from the Chaney Chemical Laboratory has been operated in the Windsor area simultaneously with the modified Owens instrument for purposes of comparison. The results of a series of runs are indicated in Table I. When the level of particulate pollution was not too high, the average concentration in mg per cu m obtained by the Owens sampler agreed fairly well with the 24-hr sample collected by the autosampler. At higher aerosol concentrations the Owens values tended to rise considerably above those of the Hall instrument. This was probably due to the small size of the spots obtained by the Owens method and the difficulty encountered in accurate evaluation of such small stains by color comparison when the mass of deposit was considerable.

Various simpler models of continuous filtration apparatus operating on the above principle have been constructed by Hemeon of the Mellon Institute (15), Von Brand,[4] and Wilson.[4] The intensity of the spots which are usually about 1 in. in diameter are evaluated by transmission or reflectance measurements. It has been found that the reflectance is almost a linear function of the amount of aerosol deposited in a given location in the range of 100 to about 50 per cent. Recently Hemeon (16) proposed a scale of transmission units called "Coh" units in which the concentration of the deposit would be expressed as a certain number of "Coh" units per 1000 linear feet of air sampled. Hall has shown that a linear relationship exists between this scale and the Hall K_{m} value.[5]

TABLE I.—DUST CONCENTRATIONS.

Time		Chaney Autosampler		Owens Filter (Shade Scale), mg per cu m
		Trans-mission, per cent	Optical Density	
WEIGHT SAMPLE—11 A.M., FEB. 16, 1953 TO 11 A.M., FEB. 17, 1953. CONCENTRATION 0.241 MG PER CU M				
Feb. 16:				
1100	No. 1.......	74.5	0.128	0.471
1200	No. 2.......	76.5	0.117	0.248
1300	No. 3.......	78.3	0.106	0.198
1400	No. 4.......	78.2	0.107	0.248
1500	No. 5.......	83.2	0.085	0.248
1600	No. 6.......	73.6	0.133	0.397
1700	No. 7.......	74.7	0.127	0.397
1800	No. 8.......	82.7	0.083	0.148
1900	No. 9.......	85.0	0.071	0.248
2000	No. 10.......	71.6	0.145	0.471
2100	No. 11.......	80.8	0.093	0.297
2200	No. 12.......	82.2	0.086	0.198
2300	No. 13.......	85.0	0.071	0.198
Feb. 17:				
0000	No. 14.......	82.0	0.086	0.248
0100	No. 15.......	86.8	0.062	0.099
0200	No. 16.......	87.2	0.060	0.099
0300	No. 17.......	88.3	0.054	0.099
0400	No. 18.......	86.3	0.064	0.198
0500	No. 19.......	87.4	0.059	0.198
0600	No. 20.......	87.5	0.058	0.099
0700	No. 21.......	86.6	0.063	0.148
0800	No. 23.......	86.2	0.065	0.099
0900	No. 24.......	83.9	0.077	0.148
1000	No. 25.......	88.3	0.054	0.075
Average........................			0.086	0.221
WEIGHT SAMPLE—11 A.M., FEB. 17, 1953 TO 11 A.M., FEB. 18, 1953. CONCENTRATION 0.254 MG PER CU M				
Feb. 17:				
1100	No. 1.......	78.5	0.105	0.198
1200	No. 2.......	79.8	0.098	0.198
1300	No. 3.......	87.6	0.057	0.075
1400	No. 4.......	84.4	0.074	0.075
1500	No. 5.......	85.2	0.069	0.148
1600	No. 6.......	86.3	0.064	0.075
1700	No. 7.......	89.8	0.046	0.050
1800	No. 8.......	86.0	0.065	0.148
1900	No. 9.......	86.0	0.065	0.148
2000	No. 10.......	84.8	0.071	0.148
2100	No. 11.......	82.8	0.082	0.248
2200	No. 12.......	80.3	0.095	0.248
2300	No. 13.......	77.5	0.110	0.297
Feb. 18:				
0000	No. 14.......	77.8	0.109	0.297
0100	No. 15.......	78.0	0.108	0.297
0200	No. 16.......	76.0	0.119	0.297
0300	No. 17.......	78.0	0.108	0.297
0400	No. 18.......	77.0	0.113	0.297
0500	No. 19.......	78.8	0.103	0.397
0600	No. 20.......	76.0	0.119	0.347
0700	No. 22.......	74.0	0.130	0.471
0800	No. 23.......	75.5	0.122	0.471
0900	No. 24.......	76.4	0.117	0.342
1000	No. 25.......	79.0	0.102	0.198
Average........................			0.094	0.240
WEIGHT SAMPLE—11 A.M., FEB. 18, 1953 TO 11 A.M., FEB. 19 1953. CONCENTRATION 0.342 MG PER CU M				
Feb. 18:				
1100	No. 1.......	80.4	0.095	0.322
1200	No. 2.......	78.4	0.104	0.213
1300	No. 4.......	77.5	0.111	0.322
1400	No. 6.......	82.4	0.084	0.213
1500	No. 7.......	81.4	0.090	0.268
1600	No. 8.......	77.0	0.125	0.322
1700	No. 9.......	79.9	0.098	0.213
1800	No. 10.......	78.8	0.103	0.322
1900	No. 11.......	77.0	0.119	0.322
2000	No. 12.......	72.1	0.142	0.370
2100	No. 13.......	76.2	0.118	0.417
2200	No. 14.......	74.0	0.130	0.417
2300	No. 15.......	77.5	0.111	0.322
Feb. 19:				
0000	No. 16	76.3	0.118	0.370
0100	No. 17.......	78.3	0.106	0.213
0200	No. 18.......	79.1	0.102	0.322
0300	No. 20.......	76.9	0.114	0.315
0500	No. 22.......	72.4	0.140	0.448
0600	No. 23.......	66.8	0.175	0.990
0700	No. 24.......	64.8	0.188	0.990
0800	No. 25.......	65.6	0.183	0.834
Average........................			0.122	0.405

Considerable discussion has arisen between proponents of either transmission or reflectance as the best method of evaluating the intensity of the stains. The diffuse reflectance and transmission results of a number of samples from the Hall-Chaney autosampler are shown in

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[4] Private communication, 1952.

[5] Private communication, 1953.

TABLE II.—COMPARISON OF REFLECTANCE AND TRANSMISSION READINGS ON PARTICULATE MATTER SAMPLES COLLECTED BY HALL-CHANEY AUTOSAMPLER.

Filter	Diffuse Reflectance	Transmission as Optical Density	$R_1 \times 0.54$
Feb. 16–17, 1953			
No. 1	0.235	0.128	0.127
No. 3	0.180	0.106	0.097
No. 4	0.205	0.107	0.111
No. 5	0.180	0.085	0.097
No. 7	0.240	0.127	0.130
No. 8	0.178	0.083	0.096
No. 9	0.154	0.071	0.083
No. 11	0.180	0.093	0.097
No. 12	0.184	0.086	0.099
No. 13	0.151	0.071	0.082
No. 14	0.170	0.086	0.092
No. 15	0.136	0.062	0.073
No. 16	0.128	0.060	0.069
No. 17	0.126	0.054	0.068
No. 18	0.135	0.064	0.073
No. 19	0.122	0.059	0.066
No. 20	0.125	0.058	0.068
No. 21	0.134	0.063	0.072
No. 23	0.140	0.065	0.076
No. 25	0.123	0.054	0.066
Feb. 17–18, 1953			
No. 6	0.110	0.064	0.059
No. 7	0.090	0.046	0.049
No. 8	0.115	0.065	0.062
No. 9	0.122	0.065	0.066
No. 10	0.120	0.071	0.065
No. 11	0.160	0.082	0.086
No. 12	0.169	0.095	0.091
No. 14	0.210	0.109	0.113
No. 15	0.195	0.108	0.105
No. 16	0.190	0.119	0.103
No. 17	0.205	0.108	0.111
No. 19	0.186	0.103	0.100
No. 20	0.208	0.119	0.112
No. 22	0.265	0.130	0.143
No. 23	0.195	0.122	0.105
No. 24	0.208	0.117	0.112
No. 25	0.182	0.102	0.098
Feb. 18–19, 1953			
No. 1	0.172	0.095	0.093
No. 2	0.186	0.104	0.100
No. 4	0.168	0.111	0.091
No. 6	0.155	0.084	0.084
No. 7	0.160	0.090	0.086
No. 8	0.211	0.125	0.114
No. 9	0.175	0.098	0.095
No. 10	0.190	0.103	0.103
No. 11	0.191	0.119	0.103
No. 12	0.241	0.142	0.130
No. 13	0.236	0.118	0.127
No. 14	0.223	0.130	0.120
No. 15	0.183	0.111	0.099
No. 16	0.191	0.118	0.103
No. 17	0.194	0.106	0.105
No. 18	0.180	0.102	0.097
No. 20	0.195	0.114	0.105
No. 22	0.270	0.140	0.146
No. 23	0.325	0.175	0.176
No. 24	0.356	0.188	0.192

Table II. The reflectance was determined in a Beckman spectrophotometer at a constant wavelength of 400 mμ with the light reflected at angles between 35 and 55 deg, and the readings taken on the density scale. The transmission results are in fairly good agreement with those for reflectance over the density range available. However, both Wilson and Hall recommended the use of the reflectance method. Hall[5] in a recent communication has stressed the importance of a monochromatic light source at 400 mμ in measurements of this type.

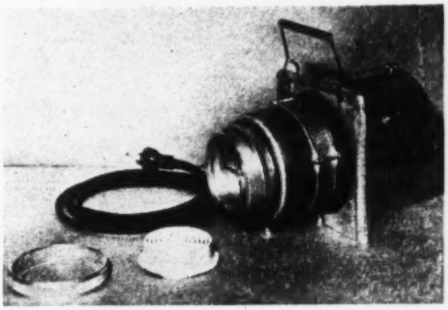

FIG. 2.—The High Volume Air Sampler.

High Volume Sampler.—The high volume air sampler has been used extensively to collect fine particulate matter in the Windsor-Detroit investigation. This sampler is shown in Fig. 2. It consists of an Electrolux blower mounted in an aluminum housing, a filter paper retaining ring located on the intake side of the blower, and a *U*-tube manometer located on the exhaust side. The manometer was calibrated with a dry gas meter to obtain the air flow in cubic feet per minute. The filter used was an accordion pleated paper (type S, unimpregnated) secured from the Mines Safety Appliances Co. The rate of flow through the average clean filter was about 65 cu ft per min. This flow decreased as the deposit of particulate matter built up on the paper, and in areas of high pollution loading would be reduced to approximately 25 cu ft per min at the end of a 24-hr sampling period. However, sufficient material could be collected over a

24-hr period not only for determination of the concentration on a weight basis but also for chemical analysis. Silverman and Viles (31) have reported collection efficiencies of 68 per cent for tobacco smoke and 99 per cent for certain other dust and fumes when collected on these filters at a flow rate of 60 cu ft per min.

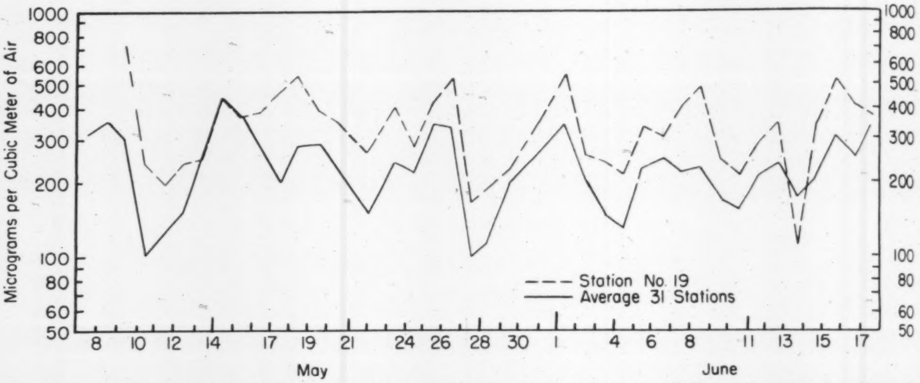

FIG. 3.—Weights of Total Particulate Matter for Daily Air Samples Collected at Station No. 19 Compared with Corresponding Averages for All Stations in the Detroit Area, May 7 Through June 17, 1951.

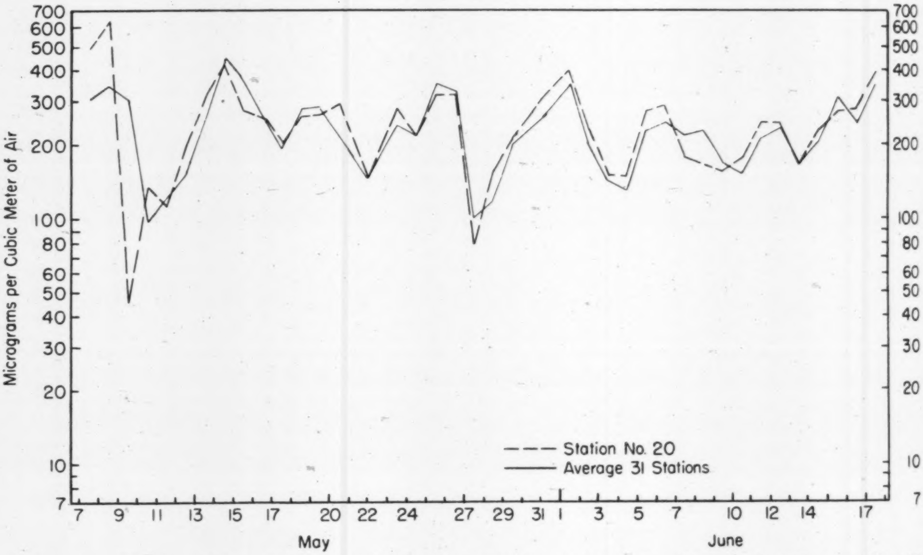

FIG. 4.—Weights of Total Particulate Matter for Daily Air Samples Collected at Station No. 20 Compared with Corresponding Averages for All Stations in the Detroit Area, May 7 Through June 17, 1951.

Daily samples (24 hr) have been collected for considerable periods at 25 stations in the Windsor area and 31 stations in the Detroit area. The methods of weighing and spectrographic analysis are described in a paper by Keenan and Byers (23). Some of the results for Windsor have been presented by Katz elsewhere (20, 21). The results obtained by Clayton for the Detroit area in a representative run from May 7 to June 17, 1951, are illustrated in Figs. 3 and 4. They show the daily variations in the average for 31 stations compared with station 19, which showed fluctuations generally higher than the average of all stations, and station 20, which followed the average daily variations at all stations fairly closely.

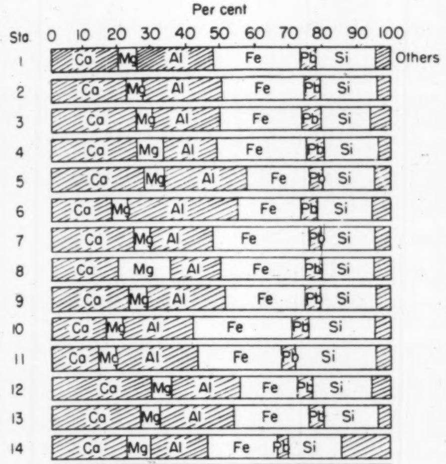

FIG. 5.—Distribution of Ca, Mg, Al, Fe, Pb, and Si in 24 Elements of Fine Particulate Matter at Sampling Stations in Detroit Area.

A considerable number of samples collected as above were analyzed spectrographically for 24 elements. The main contribution to the inorganic fraction of the particulate matter was due to six elements, namely, calcium, aluminum, iron, silicon, magnesium, and lead. The average contribution in per cent of these six elements to the total weight of the 24 elements at each of the 31 sampling stations over the above test period in the Detroit area is shown in Fig. 5.

Samples collected with the high volume sampler were also analyzed chemically for chlorides, fluorides, and sulfur. The chlorides were determined by the method of Caldwell and Mayer (2), and fluorides by the method of Talvitie (35). The high volume samples probably yield solid chlorides and fluorides only. Samples for these compounds were also collected with the standard impinger. The data in Table III indicate that the average value for chlorides in the Detroit area collected by the standard impinger was approximately six times the average value for samples collected with the high volume air samplers. In the case of fluorides there was approximately a sevenfold increase in the average value obtained with the impinger to the average value obtained with the high volume air sampler. The difference between the values obtained by these two methods is probably due to the gaseous fluorides and chlorides present in the atmosphere.

Size Distribution.—The particle size distribution of the air-borne particulates in the Detroit area is indicated by the cumulative curves shown in Fig. 6 for nine standard impinger samples and twelve thermal precipitator samples. With the standard impinger 87 per cent of the samples were less than 2 μ in size. With the thermal precipitator, 83.9 per cent of the samples were below 2 μ in size. The thermal precipitator demonstrated a greater collection efficiency than the standard impinger in the size below 0.50 μ.

TABLE III.—RELATIONSHIP OF CHLORIDE AND FLUORIDE VALUES OBTAINED WITH THE STANDARD IMPINGER TO CHLORIDE AND FLUORIDE VALUES OBTAINED WITH THE HIGH VOLUME AIR SAMPLER.

All values are given in micrograms per cubic meter of air.

Station	CHLORIDES				FLUORIDES			
	Filter Paper Samples Average	Impinger Samples[a]	Number of Impinger Samples	Number of Filter Paper Samples	Filter Paper Samples Average	Impinger Samples[a]	Number of Impinger Samples	Number of Filter Paper Samples
No. 1	1.34	8.82	2	6	0.17	0.15	2	6
No. 2	1.41	2.94	1	5	0.13	0.39	1	5
No. 3	0.80	...	0	6	0.09	2.45	1	6
No. 4	1.32	...	0	6	0.15	0.86	2	6
No. 5	0.95	5.88	1	6	0.07	1.51	3	6
No. 6	1.01	10.13	2	6	0.09	0.10	1	6
No. 7	1.29	7.84	3	6	0.17	0.57	4	6
No. 8	1.24	5.88	1	6	0.08	0.10	1	6
No. 9	1.21	2.94	1	6	0.07	2.42	2	6
No. 10	1.12	11.90	1	6	0.11	0.68	1	6
No. 11	1.11	2.94	1	6	0.11	0.38	1	6
No. 12	0.39	...	0	5	0.06	1.21	2	5
No. 13	1.00	2.94	2	6	0.07	0.29	2	6
No. 14	1.09	...	0	6	0.24	...	0	6
No. 15	1 74	5.88	2	6	0.27	1.04	3	6
No. 16	1.02	17.64	1	6	0.17	0.00	1	6
No. 17	0.85	2.65	2	6	0.10	1.61	2	6
No. 18	0.71	...	0	6	0.07	3.47	1	6
No. 19	2.18	5.88	1	6	0.40	0.50	2	6
No. 20	1.36	4.83	1	6	0.27	2.84	2	6
No. 21	0.92	...	0	5	0.08	...	0	5
No. 22	0.69	...	0	6	0.07	...	0	6
No. 23	0.46	2.94	2	6	0.09	0.10	2	6
No. 24	0.69	2.94	2	6	0.11	0.24	2	6
No. 25	0.91	8.82	1	5	0.06	0.10	1	5
No. 26	1.53	...	0	6	0.10	0.10	1	6
No. 27	0.39	3.45	2	6	0.05	0.00	1	6
No. 28	0.53	7.35	2	6	0.05	0.10	1	6
No. 29	0.51	...	0	6	0.05	...	0	6
No. 30	1.08	...	0	5	0.13	1.94	2	5
No. 31	1.18	2.94	1	6	0.09	0.46	3	6
Average	1.03	6.07			0.12	0.87		

[a] Where more than one sample is indicated the average of the samples was used.

It is evident from a consideration of the nature of the deposit produced when polluted air is drawn through filter paper that the intensity of the stain or spot produced must bear some relation to the mass concentration of the particulate matter in unit volume of air. This consideration is valid only if the sample of particulate matter is truly representative of the general pollution in the area which gives rise to haze or reduced visibility and if the sampling station is not too close to some local source of fly ash or large dust particles. In order to provide some test for this hypothesis, the particle size distribution of the dust collected on some Owens filter samples in the Windsor area was determined and an estimate made of the mass concentration from the number count.

The most convenient method of counting particles embedded in filter paper is with the stereoscopic microscope. This, however, has a limit of resolution of approximately 1.0 μ, thus preventing the inclusion of a large number of very small particles. To overcome this restriction, a number of fibers were carefully extracted from a test stain, and all the particles on these were counted with both the high-power monocular and the stereoscopic microscopes. By a comparison of distribution, a correction could be applied to the second count to include particles down to approximately 0.5 μ.

Two test stains of concentrations, 0.065 and 0.120 mg per cu m, were selected from the Owens filter samples and the excess filter paper carefully pared off to increase the transparency. Higher concentrations could not be counted because of the superposition of particles. Counts were made on these under the stereoscopic microscope utilizing a Whipple disk.

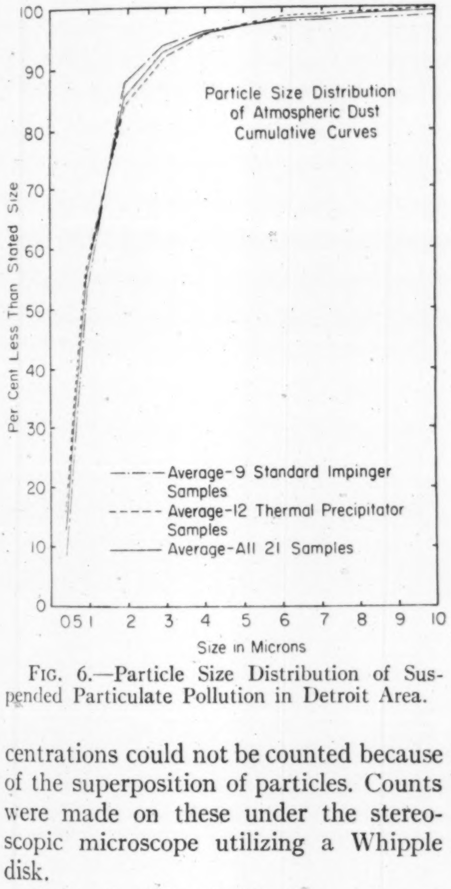

Fig. 6.—Particle Size Distribution of Suspended Particulate Pollution in Detroit Area.

The probability distribution of sizes of particulates is a skewed curve which can be normalized by expressing the sizes as logarithms. It was observed that the curves for the fibers fell off more rapidly for large sizes than did those for the stains. This may have been due to the loss of some of the larger particles when single fibers were extracted from the filter paper.

By weighting the size frequency for each class in terms of the cube of the average size value representing that class, plotting in the same manner as the size-count analysis the size mass distribution is obtained.

Assuming a density of 1.0 for the particulate matter, the concentration may also be obtained. The mass factor was calculated on the assumption of spherical particles and is in units of 10^{-9} mg.

Total Numbers and Mass of Particulates Collected

Diameter of stain....... = 3.14 mm
Area of stain........... = 7.744 sq mm
Area counted........... = 0.56 sq mm
Total number of particles on stain I............ = 806 × 13.83 = *11,147*
Total mass of particles (30 l air)............ = 119,416 × 13.83 × 10^{-9} mg
= 1.6515 × 10^{-3} mg
Concentration.......... = *0.055* mg per cu m
Concentration estimated from shade scale...... = *0.065* mg per cu m
Total number of particles on stain II........... = 1345 × 13.83 = *18,601*
Total mass of particles (30 l air)............ = 312,868 × 13.83 × 10^{-9} mg
= 4.327 × 10^{-3} mg
Concentration.......... = *0.144* mg per cu m
Concentration estimated from shade scale...... = 0.120 mg per cu m

Hall's K_m values for a number of Owens filter paper samples were calculated after obtaining the transmission and density readings for the stains, as shown in Table IV. Differences in concentration between samples which cannot be detected by means of the Owens shade scale become apparent when the spots are evaluated by a more accurate optical technique.

Sulfuric Acid Aerosol Analyzers.—The

Stanford Research Inst has developed a continuous aerosol analyzer in which the particles are deposited by an electrostatic precipitator on a rotating disk of stainless steel which dips into a stream of water (**1**, **29**). This instrument is used to measure sulfuric acid aerosol continuously in a conductivity cell of small volume. The instrument would be useful for other types of aerosols that are soluble in water and form an electrolyte.

Thomas (**36**) has used an impactor type analyzer for several years. It consists of a glass impactor with a slit 0.125 × 8 mm communicating with a conductivity cell. Suction is applied to maintain about 0.3 atmosphere pressure at the slit. The acid is collected by impaction at sonic velocity on a glass surface and is periodically washed off with a measured volume of water. The acid aerosol concentration is determined by recording the conductivity of the resulting solution, even in the presence of sulfur dioxide of a hundredfold concentration.

TABLE IV.—K_m READINGS FOR OWENS FILTER PAPER SAMPLE.

PHOTOMETER READINGS

Samples	Concentration (from shade scale)	Transmission Reading	Log 100/T	(−0.037)	K_m	Carbon Concentration,[a] mg per cu m
No. 1 Clean filter A	0.0	92.5	0.034	−0.003		
No. 2 Jan. 23 1952 1300	0.088	54.0	0.268	0.231	5.45	0.057
No. 3 Jan. 23 1952 1330	0.088	51.0	0.293	0.256	6.04	
No. 4 Jan. 23 1952 1400	0.120	45.0	0.347	0.310	7.32	0.076
No. 5 Jan. 23 1952 1430	0.120	43.0	0.367	0.330	7.79	
No. 6 Jan. 22 1952 1000	0.150	35.0	0.456	0.419	9.89	0.107
No. 7 Jan. 22 1953 1030	0.153	30.0	0.523	0.486	11.47	
No. 8 Jan. 22 1952 1330	0.210	25.0	0.602	0.565	13.33	0.131
No. 9 Jan. 22 1952 1400	0.210	26.0	0.585	0.548	12.93	
No. 10 Clean filter B	0.0	91.0	0.041	+0.004		

DENSITOMETER READINGS OF SAME SAMPLES

	Reading	(× 0.04) Density	(−2.69)	K_m	Carbon Concentration,[a] mg per cu m
No. 1	66.5	2.66	−0.03		
No. 2	70.5	2.82	0.13	3.1	0.041
No. 3	72.5	2.90	0.21	5.0	
No. 4	74.5	2.98	0.29	6.8	0.073
No. 5	75.5	3.02	0.33	7.8	
No. 6	77.2	3.09	0.40	9.4	0.105
No. 7	79.5	3.18	0.49	11.6	
No. 8	81.8	3.27	0.58	13.7	0.131
No. 9	80.6	3.22	0.53	12.5	
No. 10	68.0	2.72	+0.03		

K_m = that deposit which produces an optical density of 0.1 when the deposit area is 1 sq cm and the volume of air sampled is 1 cu m.

Area of test spot (diameter 0.3 cm) = 0.0707 sq cm.

Volume of air sampled = 30 litres = 0.03 cu m.

$$K_m = De \times 10 \times \frac{0.0707}{0.03} = 23.6 \times De$$

[a] According to Hall (**14**) the carbon content for the Los Angeles district is approximately equal to 10 micrograms per cubic metre for unit K_m (based on reflectance measurements).

These readings have been taken beyond the range of linearity which is 0.05 to 0.3 in optical density.

Light Scattering Instruments.—A recent paper by Sinclair (**33**) describes the design and preliminary calibration of a light scattering instrument for the measurement of the mass concentration or dust loading of the atmosphere. It is designed to operate a recording potentiometer for continuous outdoor measurement wherever 60 cycle AC is available. In instruments of this type, according to Sinclair, the aerosol concentration is commonly measured by three methods: (1) transmission, (2) right-angle scattering, and

(3) forward scattering. Of these, forward scattering is the most practical method to provide a sufficiently sensitive instrument, with a small light path, for most atmospheric aerosols. Other developments in this field have been described by Gucker *et al*, (12, 13), Sinclair and La Mer (32). O'Konski, who has been associated with Gucker for many years in research on measurement of aerosols by light-scattering methods, is planning to build a model for general outdoor pollution work in the near future in California. It must be understood that light-scattering measurements *per se* cannot distinguish quantitatively between concentration and particle size when there is present a considerable range of sizes. Ordinarily the particle size range of suspended matter in the atmosphere may vary as much as a hundredfold or more. However, much useful information can be obtained by the measurements if supplemented by other collection methods such as filtration and dustfall.

Visibility Meters.—A number of cameras or photoelectric photometers have been developed which measure the apparent contrast ratio for black targets against the horizon sky. In the Steffans camera (34), photographs are obtained of black targets at various distances and the visibility is then computed from the relative density of the images. The Chaney instrument (4) is a recording visibility meter which utilizes two black targets at different distances from a photoelectric photometer. The two targets are viewed in turn through different apertures varied by a slow-speed motor-driven diaphragm. The visual range may be calculated from the chart readings. More recently Marynowski and Littman (26) of the Stanford Research Inst. have described a continuous recording visibility meter which consists of a photoelectric photometer aimed at a dark target, a photovoltaic cell exposed to an artificial horizon and a modified Leeds & Northrup "Micromax" recorder. In practice, the dark target is a black gas holder located 1000 ft away in a nearly southerly direction so that its face is not illuminated by direct sunlight.

Most visibility instruments can be operated in daylight only. However, the transmissometer records visibility continuously day or night, an important factor in air pollution studies. The Transmissometer has been operated experimentally in Detroit for the past ten months. The instrument was developed by C. A. Douglas (9) of the National Bureau of Standards and is an electronic device which measures accurately and continuously the light transmission between two fixed points. The instrument was designed to aid in flying safety through eliminating or reducing the human factor in visual estimations.

The transmissometer consists of a 350,000 candle power light source, a phototube receiver, an amplifier, and an indicator. The distance between the points of transmission and reception is about 800 ft. The output of the receiver is transmitted to the indicator and the recorder which may be located several miles from the actual site of the testing.

The intensity of the light is controlled by rheostats and an automatic cutoff is made hourly for zero checks. Zero checks may also be made at the recorder as indicated or desired. The expected service life of a lamp is from three to six months.

The receiver unit consists of a lens, a diaphragm, a photo-pulse unit, and an amplifier. These are shown in the photograph, Fig. 7. The light focused by means of the lens, on a pinhole in the diaphragm, strikes the phototube receiver and there generates pulses whose frequency is directly proportional to the intensity of the light on the phototube. These pulses are amplied and transmitted to the indicator.

In the pulse generator unit a phototube sets up a current which charges a capacitor with sufficient voltage across it to cause a discharge through a neon lamp. The capacitator rapidly discharges through the neon lamp and a resistor until the voltage is no longer sufficient to maintain a current through the lamp. The voltage drop across the resistor supplies a voltage pulse to the grid of a 6J5 tube causing a momentary change in the plate current of the tube. The resulting momentary change in voltage drop across a transformer when amplified supplies the pulse signal which is received at the indicator.

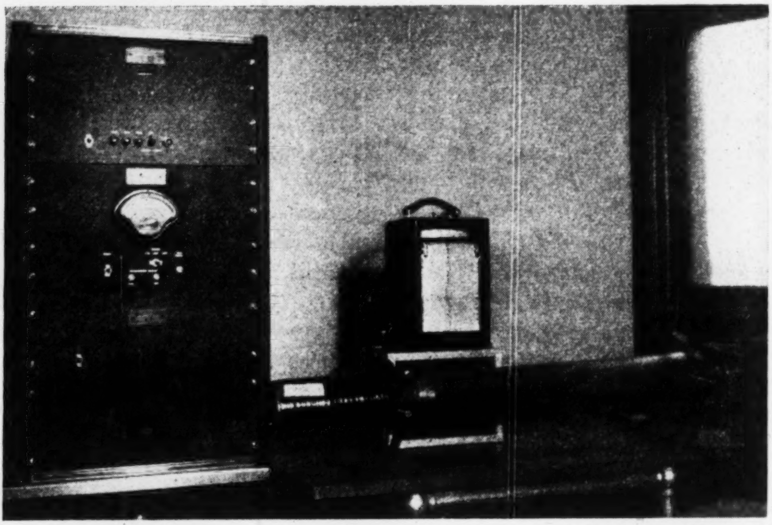

FIG. 7.—Receiver Unit of Transmissometer Showing Long Brass Tube Containing Lens and Diaphragm, Photo-pulse Unit and Chart Recorder.

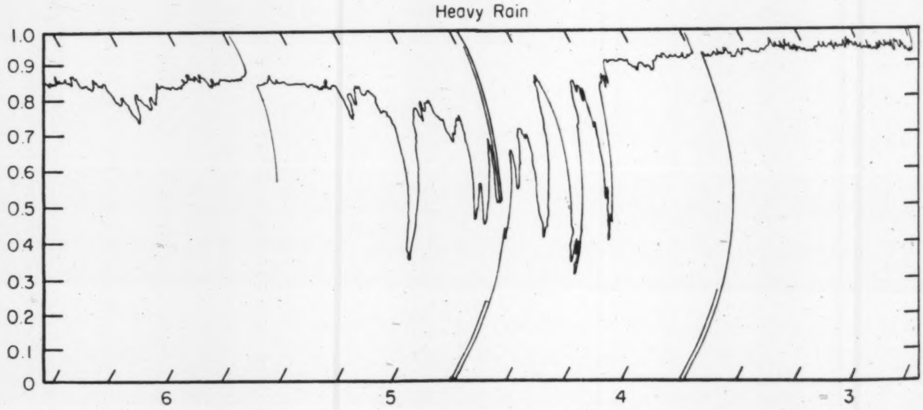

FIG. 8.—Sample Chart Record of Transmissometer Showing Effect of a Short Period of Heavy Rainfall. Ordinates Represent Transmission Units, Abscissae Represent Time in Hours.

The indicator consists of a frequency measuring unit, a 2-stage amplifier, and a calibrator. The frequency unit levels and averages the pulses which are received through the amplifier and produces a meter reading which is directly proportional to the pulse frequency and therefore to the transmission of the atmosphere between the receiver and the light source.

A recorder connected to the indicator gives a continuous record of the indicator meter reading on chart paper marked to show percentage of light transmission by time of day. A sample chart record is shown in Fig. 8.

In the original test work on this instrument, nearly 3000 simultaneous observations of the visual range and transmissometer readings were made. These data were used to plot curves for transmission and visual range for settings of varying distances between the fixed points. From these curves calibration of the transmissometer for any distance up to $1\frac{1}{2}$ km can be made.

The instrument was operated at fixed points 250 m apart at an elevation of approximately 125 ft above the ground. Little distortion of light due to heat radiation was noted and the instrument proved to be extremely sensitive to even minor variations in light transmission. With experience in viewing the charts, smoke clouds and meteorological phenomena can be recognized.

Tests are now being conducted to determine the usefulness of this instrument in the field of air pollution research. It is the ultimate desire to calibrate this instrument in terms of suspended particulate matter. This will eliminate the tedious and costly laboratory and field techniques currently in practice. In the evaluation of the instrument, consideration is given to the separation of natural *versus* industrial air pollutants, that is, fog particles *versus* visibility-reducing contaminants.

GASES

It is beyond the scope of this paper to deal with continuous instrumentation and analytical techniques for all the gaseous compounds likely to be found in the polluted air of cities or industrial areas. Only a brief outline of some of the more important practical methods dealing with sulfur dioxide, hydrogen sulfide and other sulfur-containing gases, hydrogen fluoride, and "smog gases" will be attempted here.

Sulfur Dioxide:

Thomas (**40, 41**) of the American Smelting and Refining Co. has pioneered the development of various types of continuous analyzers for the determination of low concentrations of sulfur dioxide. The Thomas autometer absorbs the gas in slightly acidulated (0.00002 N) distilled water containing hydrogen peroxide (0.002 M) and measures the electrolytic conductivity of the resulting sulfuric acid solution. A continuous record of the conductivity is obtained by means of a recording Wheatstone bridge galvanometer. The volume flow of air through the absorbers is also recorded. Both "accumulating" and "instantaneous" types of instruments are available. In the former type, the trace record indicates the total change in conductivity over a sampling cycle of 30 min, which is a measure of the average concentration. Any variations in the shape of the conductivity curve indicate variable concentrations during the sampling period, so that short-period peaks can also be evaluated from the trace. The gas is absorbed in glass scrubbers of the bubbling type, with the electrodes placed below the bubbler so that gas bubbles in the solution do not reach them.

In the instantaneous type of analyzer, a regulated flow of solution passes down along a metal spiral wire on the inside wall of a long glass tube, countercurrent to the air stream. The ratio of gas flow to liquid volume may be as high as 5000 to 1. The liquid after absorbing the sulfur dioxide flows through a small conductivity cell, volume about 0.3 ml. This provides a continuous short-period record of the concentration or a 1- to 2-min average. The average concentration over a 30-min period is also indicated by means of an accumulating cell.

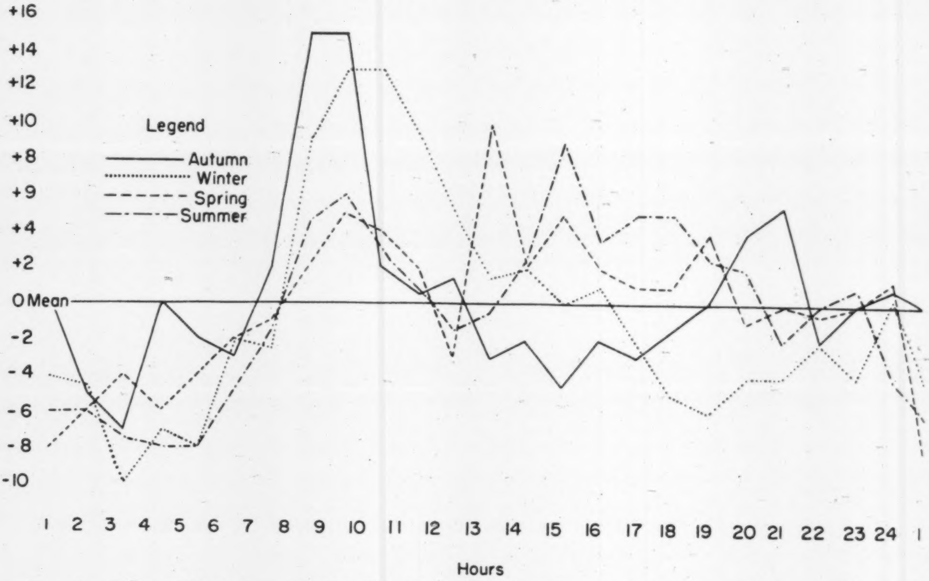

FIG. 9.—Diurnal Variation of Sulfur Dioxide in Windsor, Ontario, for Various Seasons of the Year

The various types of autometers are illustrated in a recent paper by Thomas (36). Studies of the sulfur dioxide content of the atmosphere in smelter areas and elsewhere over many years by Thomas, Katz (17, 21) and others have demonstrated the usefulness of this type of equipment. However, the Thomas autometer has its limitations when employed to analyze the complex city air. It is sensitive to any soluble gas or aerosol which may produce an electrolyte in solution such as ammonia, hydrochloric acid, or other halides, sulfuric acid, or lime dust. Some of the limitations of this instrument have been discussed by Perley and Langsdorf (28) and by Giever (11). Nevertheless, its use is almost indispensable in air pollution studies, especially in areas where the amount of sulfur dioxide emitted to the atmosphere is known to constitute a major source of pollution.

A number of these instruments have been employed continuously in the Windsor-Detroit area where the annual consumption of solid fuel alone is about 16 million tons and the estimated over-all emission of sulfur dioxide is greater than 430,000 tons per annum. The highest sulfur dioxide concentrations were found in industrial and residential areas lying close to the Detroit River. Comparisons are available in Windsor between three test stations within the city and a rural station 15 miles south of the Detroit River, over a period of several years. The data indicate widespread distribution of sulfur dioxide pollution during occasional periods. Thus in September, 1952, a 30-min maximum of 1.45 ppm was recorded at one of the stations close to the river. The maximum concentration at the rural station reached a value of 0.876 ppm. Normally the maximum concentration at the rural station does not exceed 0.25 ppm. The highest monthly mean concentration within the city area reached a value of 0.20 ppm in December, 1952. The curves in Fig. 9 illustrate the diurnal variation in sulfur dioxide for different seasons of the year at a station in Windsor over the period September, 1950 to January, 1953. The rise in concentration in the morning period of the day is most pronounced in the autumn and winter seasons.

The "titrilog" is another instrument which has been used successfully for the continuous determination of sulfur dioxide (**40**). The air sample is passed through a bromide solution by means of a gas bubbler in an electrolytic cell which contains an inner "sensor" electrode and a bromine generator electrode. The liquid is circulated through the cell by the gas stream. If sulfur dioxide or any other oxidizable gas is present, an equivalent amount of bromine is produced under the control of the "sensor" electrode. The concentration of bromine in the inner cell produces a voltage on the sensor electrode which is connected to the input of an amplifier. The amplifier is so adjusted that when the sample is free from oxidizable gases only a minimum amount of bromine is generated corresponding to the zero line on the recorder chart. As the electrolyte circulates, it passes through a carbon filter which removes any impurities flowing out of the inner cell.

The titrilog is sensitive to hydrogen sulfide and organic sulfides and, to a lesser extent, to unsaturated hydrocarbons. A reverse reaction takes place with nitrogen peroxide and chlorine. Thomas has shown simultaneous records obtained with the titrilog and the autometer at Los Angeles (**36**), using a common intake. The two instruments agreed generally in the observations but at times gave appreciably divergent results.

Katz (**18**) has described a photoelectric recording apparatus for sulfur dioxide based on the use of dilute solutions of potassium iodide-starch-iodine in the concentration range of 0.00007 N to 0.00002 N, with respect to iodine. The reaction with dilute sulfur dioxide causes a fading of the blue color which is measured photometrically. The instrument was operated for several seasons in the Sudbury smelter area. The instrument has a range of less than 0.01 to 1.0 ppm or more depending on the size of the gas sample and the volume of solution used in the absorbers. This method is subject to interference by hydrogen sulfide but to a lesser extent than the titrilog because the solution is much less reactive than bromine. The method is probably more specific for SO_2 than the autometer.

Hemeon has adapted his paper filtration apparatus to the semiautomatic measurement of sulfur dioxide over hourly periods. The air is filtered through a 1-in. diameter area of filter paper strips moistened with glycerine and sodium hydroxide. A fresh area of paper is exposed automatically every hour. At the end of a given period the spots are removed and extracted with water, and the solution is oxidized and analyzed for sulfate (**15**). It is evident that the method involves considerable laboratory work.

Hydrogen Sulfide and Organic Sulfides:

These gases have been determined for many years in the field by the Thomas autometer after a preliminary combustion of the sulfur compounds to sulfur dioxide by passing the gas sample over a platinum wire heated electrically to about 550 C (40). The sulfur dioxide already present in the gases is determined by the analysis of a parallel unheated gas stream. The method is sensitive to 0.01 ppm of sulfide. The titrilog instrument can also be used to determine sulfides with comparable accuracy.

Wilson[6] has constructed a paper filtration apparatus for the determination of hydrogen sulfide. This employs lead-acetate-impregnated paper strips with spots about 1 in. in diameter, advanced at regular intervals by a clock mechanism. The volume of air filtered through the paper in each position is measured, and the intensity of the spots is determined by comparison with calibrated standard spots. This method has been used by W. G. Fredrick of the Detroit Department of Industrial Hygiene. A similar method employing paper impregnated with lead acetate and glycerine, sensitive to 0.05 ppm in 1-hr test samples, is being developed by Hemeon (15).

The above gases can be readily detected by odor in extremely low concentrations as low as 0.025 ppm. Some mercaptans or organic sulfides give a perceptible odor in concentrations less than 0.01 ppm. It is evident therefore that instruments for this class of components have to be extremely sensitive to measure odor nuisances.

Filter Tape Gas Recorders:

Continuous filter tape recorders for gases are a comparatively new development. The tape or paper is impregnated with chemicals suitable for reacting with each type of gas or vapor. In the Von Brand filtering recorders, the medium under test is sampled at a uniform rate through a continuously progressing filter tape guided past a recording head of special design. Adjustment of sampling rate and selection of both tape speed and working area in the head give a very wide range of sensitivity and facilitate calibration. Recording heads are available to produce single, dual, or triple traces on the paper about $\frac{1}{4}$ in. in width, or circular spots of $\frac{3}{4}$ in. diameter.

An instrument described in a bulletin of the Vitro Corporation of America called the Microsensor detects micro quantities of gases. The instrument consists of three principal parts, a gas intake system, a reel of chemically treated paper tape, and a precision color comparator. A recording device may also be used as a part of the unit.

The gas intake system draws through the instrument a continuous sample of the atmosphere to be analyzed for a particular gaseous material. Incoming gas is divided into two channels. The first, or reference channel, contains a selected scrubber, usually activated charcoal, to filter the gas before it passes through the tape at the detector unit. Gas from the other, or sample channel, is passed directly through the tape. The two channeled flows of gas are kept exactly equal by means of flow control devices. The channels are rejoined on the other side of the tape and the gas is drawn through a dehumidifier and another scrubber before reaching the pump and being vented.

The paper tape is the vehicle for the chemical agent used in making the color comparison between the reference and sample gases. In some cases the tape is used dry, and in others it is sprayed with a liquid reagent before it reaches the detector unit. The instrument is designed to compensate for nonuniformity of the spots due to variations in thickness and porosity of recorder tape.

Color comparison is made by a precision light comparator designed around a photomultiplier tube, a flicker photometer optical system, and an electronic computing circuit. Actual comparison is accomplished by directing two equal beams of light through the two viewed spots on the tape.

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[6] Private communication, 1952.

The instrument is portable and entirely automatic. Its greatest contribution to air pollution, which is yet to be proven, may be the reputed extremely high order of sensitivity to minute quantities of many gases, such as sulfur dioxide, ammonia, hydrogen sulfide, nitrogen oxides, chlorine, ozone, and carbon monoxide.

Hydrogen Fluoride:

This gas has caused extensive damage to fruit trees, flowering plants, and other susceptible species of vegetation in extremely low concentrations in the vicinity of aluminum refineries. It is absorbed readily by forage crops and can cause fluorosis in animals feeding on such crops Concentrations of this gas in the range of several parts to 100 parts per billion in air by volume may constitute a significant degree of pollution. Until recently there was no satisfactory, completely automatic method available for the analysis of this gas because of the necessity of removing sulfur dioxide and soluble inorganic aerosols which interfere.

Thomas (35) has described a semiautomatic method whereby fluoride is determined after absorption of the air sample for periods up to three hours in the accumulating type autometer. The solution is discharged from the machine in polyethylene bottles arranged on a turntable. The sulfate is precipitated with excess barium chloride and the clear liquid after settling is decanted, adjusted to pH 3.3, and titrated with thorium nitrate, using chromeazurol-S as indicator. The sensitivity is very great, about 0.2 parts per billion, and the only compounds which would be likely to interfere are phosphates.

A continuous recording analyzer for trace quantities of hydrogen fluoride in the atmosphere has been developed recently at the Stanford Research Institute (3). The method embodied in the instrument is based on a test described by Feigl and Heisig (10) in which a metal oxinate fluoresces under ultraviolet light and the fluorescence is quenched by exposure to hydrogen fluoride. When paper impregnated with an alkaline earth oxine (salt of 8-hydroxyquinoline) is exposed to ultraviolet light, it fluoresces with a visibily bluish color. Traces of hydrogen fluoride in an atmosphere will cause the visible color emitted to be reduced.

The continuous analyzer employs a moving strip of paper impregnated with magnesium oxinate which is put in contact with the gas to be analyzed by causing it to pass through the pores of the paper. The paper is continuously exposed to ultraviolet light, and by means of suitable color filters only the visible portion of the fluorescence is directed toward a photocell. The photocell, through an amplifying system, operates a recorder.

The sensitivity of the instrument can be varied by controlling the speed at which the paper moves and thereby the size of the gas sample to which it is exposed. The greatest sensitivity is obtained at the lowest tape speeds where the exposure time is the greatest. The lower limit of sensitivity is about 30 parts of hydrogen fluoride by volume per billion parts of air.

In preparation for the field use of the instrument, a systematic study is being made of the kinds and amount of other gases that might cause interferences in field situations. Of the materials tested thus far, strong acid gases such as hydrogen chloride or sulfuric acid aerosols have been found as the most active interfering substances. These do not cause significant interferences at concentrations below 5000 parts per billion. Representative types of particulate matter have also been tested and hereto negligible interferences have been noted.

As now constructed, the instrument is sensitive only to gaseous hydrogen fluoride and not to insoluble fluorides that may be carried by the atmosphere as particulate matter.

Oxidant Recorder for Smog Gases:

In the Los Angeles area the smog conditions are characterized by a polluted air mass which causes a reduction in visibility and, quite frequently, crop damage, eye and nose irritation. According to Littman and Magill (**24**) the Los Angeles smog occurs almost invariably under conditions of 60 per cent relative humidity or lower, and is accompanied by unusually high concentrations of an oxidant which appears to be ozone. In order to study the relationship between smog occurrence and oxidant concentration, Littman and Benoliel (**25**) constructed a continuous recorder which produces a record in terms of ozone equivalents.

The oxidant recorder consists essentially of a counter current absorption tower of glass beads as the air-liquid contacting device. The air containing ozone, nitrogen oxides, or other oxidants, liberates iodine from a buffered, neutral potassium iodide solution. The light transmission of the resulting yellow solution is measured by a double-cell colorimeter at 3600 Å. The absorbing solution is regenerated by passing it through a bed of activated carbon, and re-used. The instrument is calibrated in terms of ozone equivalents in parts per 100 million by volume.

There is no doubt that ozone occurs in relatively high concentrations in the Los Angeles area, because rubber deteriorates very rapidly on exposure during smogs. However other oxidants, including ozonides and peroxidic organic compounds, are undoubtedly present in the smog gases. The latter have to be studied by more laborious procedures including collection of condensate from large volumes of air in the Shepherd trap at liquid oxygen temperature, followed by examination of the collected material by infrared or mass spectrometer methods (**30**).

METEOROLOGICAL INSTRUMENTS

No study of any air pollution problem is complete without a concurrent evaluation of meteorological factors. In a particular location the topographical features of the terrain, the proximity of land and water surfaces, and other features of the environment are a constant. The micrometeorology and the number and extent of emissions are the elements whose variations can account for the observed fluctuations in the degree of pollution. However, in a large, highly industrialized area, such as the Windsor-Detroit region, the short-term variations of emission of contaminants may be smoothed out by the averaging effect of many industries. There will be a diurnal pattern of industrial activity together with a weekly cycle, but any observed large fluctuations in the pollution level from day to day and from season to season will be related to meteorological factors.

Thomas (**36, 37**) has presented recently excellent papers on the various types of continuously recording meteorological instruments in current use in air pollution research. In his latest paper he stresses the advantages of recording the chemical and meterological data side by side on the same chart, and suggests means for accomplishing this. It is recognized that the fundamental factors which need to be measured are wind direction and velocity, turbulence of the air, and temperature and velocity gradients with height above surface, the relative humidity, precipitation and insolation, and the diffusion coefficients in the vertical and cross-wind directions.

The subject of meteorological instrumentation in this paper will be confined to a discussion of the instruments in use in the Windsor-Detroit area. The ultimate goal in this study is to formulate as precisely as possible the meteorological conditions likely to induce smog, to evaluate the relation between pollution and weather, and to isolate the influence of as many weather factors as possible on the ambient pollution.

Wind Speed and Direction:

This is being measured by Bendix Friez Aerovane recorders near surface level to correlate with simultaneous observations on sulfur dioxide and particulate contaminants. It is planned to measure the wind at 300 ft above ground as well in order to correct for surface roughness factors.

Wind Gustiness:

The aerovane records are being evaluated to show the range of variations from the mean wind speed and wind direction, or gustiness. The characteristics of the eddy motions are being studied by examination of the variability of the observed wind in order to evaluate simple gustiness parameters. This study will include not only surface observations but those at 300 ft above ground.

Temperature Lapse Rate:

Equipment is being assembled to determine the vertical temperature gradient of the atmosphere by resistance thermometers installed on a television tower at eight elevations up to about 800 ft above ground. This will yield information on the onset and duration of inversion periods, or conversely on periods of atmospheric instability.

Rainfall Measurements:

Considerable controversy exists about the effect of rain on gaseous and particulate contamination. It is not clear whether the cleansing of the atmosphere after a period of precipitation is due to the action of rain drops in removing particles suspended in air or is caused by the resultant atmospheric instability and improved turbulent mixing. The levels of gaseous and particulate pollution in the Windsor-Detroit area are being correlated with data from a rainfall recorder in order to throw more light on this point.

Illuminometer:

The total incoming daylight, both direct and reflected, in the visible light range will be measured simultaneously by recording illuminometers which are being installed at a station in downtown Windsor and at a location in a rural area 15 miles distant. The data will indicate the reduction in sunlight caused by smog conditions in downtown Windsor in comparison with conditions in the surrounding countryside.

CONCLUSION

It is apparent that continuous instrumentation and analytical techniques in air pollution are still in an early stage of development. Apart from a few gases such as sulfur dioxide, hydrogen sulfide, and hydrogen fluoride, instrumentation methods are decidedly limited. Most of the continuous methods used for gaseous pollutants are not specific for a particular type of compound. The measurement of hydrogen fluoride by the degree of quenching of the fluoresence of alkaline earth oxines is a novel approach. Perhaps the future will see the application of polarographic methods which may be made specific for certain gases.

With aerosols the best that can be done with most of the present methods is to collect the aerosols on a continuous basis and analyze for certain chemical constituents separately. Much can be learned from the evaluation of the mass concentration, particle size distribution and chemical identification of a portion of the complex materials. In this connection X-ray diffraction is proving a valuable tool in the identification of crystalline material present in the atmosphere of the Windsor-Detroit area.

For the study of the effects of air pollutants on public health and vegetation, continuous instrumental methods are indispensable. A study of public health has been in progress in the Windsor-Detroit area for some months, based on sample populations from areas of high and low pollution. During this study the environment is being assessed on a continuous basis by sampling for dustfall, suspended particulate matter, and gaseous pollution. The work is supplemented by chemical analysis and identification of as many major pollutants as possible.

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DISCUSSION

Mr. G. M. Rapp.[1]—Would Mr. Katz describe briefly the principal features of the thermal precipitator collector, such as the plate gap and the temperature head. It is my understanding that these devices are not commercially available in this country, and it may be presumed that he designed his own.

Also, what was the basis of the indicated good agreement between the thermal precipitator and the impinger device. Was it efficiency, percentage of total particulate matter removed, comparison on a weight basis, or absolute count?

Mr. Morris Katz (*author*).—The thermal precipitator is based on the principle that in a dust-laden atmosphere there is a dust-free space surrounding a hot body. The hot body is a horizontal wire of small diameter mounted in a comparatively massive block of brass, called the precipitator head, having two horizontal holes in which are inserted microscope cover-glasses. These cover-glasses are disposed vertically on either side of the wire so that they lie partially within the dust-free space. The air is drawn slowly through the channel past the wire by means of a water aspirator equipped with a calibrated glass jet, at a rate of about 50 to 100 cu cm per min. The dust particles, being unable to penetrate the space surrounding the hot wire, are deposited in a line parallel to the wire on each cover-glass. The wire is heated electrically by current from a battery connected to the head. Particles from 5 μ down to the smallest visible optically are deposited with an efficiency approaching 100 per cent.

The work on the size distribution of air-borne particulates was done on an absolute count basis. The size of particles counted are in general limited down to half a micron. The limit of optical visibility with a high-power light microscope is about 0.2 μ, but it is difficult to count such particles accurately.

As indicated in the paper, using standard counting technique, there was good agreement between the size distribution curves with samples collected by the thermal precipitator and the standard impinger, in the size range down to 0.5 μ. Therefore, in practical field work, the standard impinger will yield amost as accurate results as that obtainable with the more expensive thermal precipitator. For particles less than 0.5 μ in size the latter instrument has the higher efficiency.

The thermal precipitators were purchased from C. F. Casella & Co. Ltd. of London, England.

Mr. Rapp.—Is the author familiar with the thermal precipitators used in South Africa in the diamond mines? They apparently made their own.

Mr. Katz.—I think at that time the instrument was not available commercially.

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[1] John B. Pierce Foundation, New Haven, Conn.

If it was later than 1945, however, this thermal precipitator would have been available.

MR. W. J. SMITH.[2]—I am familiar with the instrument as made by Casella. There is also an instrument made by Joseph B. Ficklen, of Pasadena, Calif.

We made our own thermal precipitator following the description given by the original designer; ours works very satisfactorily. Making it was really not difficult.

MR. LLOYD L. FALK.[3]—Are any of the instruments described by the author suitable for ASTM to recommend as a standard instrument for air pollution work?

MR. KATZ.—I believe the Hall-Chaney instrument has been described and tested sufficiently to be recommended to the ASTM as one of the instruments to be used in atmospheric pollution studies for fine, suspended particulate matter.

There are other instruments which I mentioned in my paper; for instance, there is a paper filtration instrument developed by Wilson; there are a few others, like tape-recording instruments, which are appearing on the market rapidly. The advantage of the Hall instrument is that it not only provides a means of assessing the intensity of the particulate pollution, by optical methods such as reflectance or transmission, but it also yields a 24-hr sample for chemical analysis and weight concentration. I think this feature places it far above all others in that class.

MR. DAVID L. FRY.[4]—I would like to ask Mr. Katz whether he has noticed any pollution in the Detroit-Windsor area directly attributable to automotive engines. There has been considerable discussion in Los Angeles about air pollution due to automotive engines—the unsaturates causing harmful effects to people, plants, and farms.

MR. KATZ.—I presume this question refers to the eye irritation, farm crop injury, and so on, caused by the oxidant properties of these smog gases.

In the Windsor-Detroit Area, there was quite a serious episode of eye irritation in September, 1952. We have not yet evaluated the crop damage effects. An investigation is in progress on possible crop damage this summer.

As for the oxidant properties of the air in the Los Angeles area—I do not think we have anything like that in the Windsor-Detroit Area. There is a unique and relatively high oxidant, or ozone, concentration in the Los Angeles area. The eye-irritating properties of smog have been noted in the Windsor-Detroit Area, and in several other polluted communities. I think this is probably one of the more general properties of a polluted atmosphere.

Those of you who have read a recent paper by Mr. Magill of Stanford Research Institute will remember that he made up a synthetic gas mixture of oxides of nitrogen and some hydrocarbons, gasoline vapor, sulfur dioxide, and a few other compounds. This mixture had marked eye-irritant properties when tested in a gas chamber.

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[2] Chemical Engineer, Arthur D. Little, Inc., Cambridge, Mass.

[3] Waste Consultant, E. I. du Pont de Nemours and Co., Wilmington, Del.

[4] Supervisor of Spectroscopy, General Motors Corp., Detroit, Mich.

VARIABLES IN MONTHLY DUST FALL MEASUREMENTS*

By J. Deane Sensenbaugh[1] and W. C. L. Hemeon[1]

Air pollution by solid particles may be classified in two categories according to their pollution effect: Fine particles tend to deposit on wall and ceiling surfaces and are responsible for smoke and haze effects. Coarse particles, on the other hand, are subject to gravity deposition and constitute the quality of air pollution referred to as "dust fall," analogous to the term "rainfall." The present discussion is confined to the latter.

Just as it is logical in weather observations to record the quantity of rain reaching the ground rather than the quantity of water in temporary suspension in the atmosphere, so it is logical to measure the rate of gravity deposition of dust to obtain a record of this kind of air pollution. A weight measurement of solids suspended in the atmosphere is no more logical and is far more complicated and expensive.

Dust fall measurements are made commonly on a monthly basis by exposing open-top jars to the atmosphere in different localities and weighing the deposit. Its purpose is to provide a basis for comparison over long time periods of changes in dust fall, for comparisons between districts within a city, or for comparisons between cities.

It is often argued that the measurement lacks significance or that comparisons between cities are not permissible for various reasons. We have carried out a broad exploration of the variables involved in dust fall measurements over the past two years, the results of which are presented in the following discussion.

Variables in Dust Fall Measurements

Significance of Units:

The results are usually expressed in the units tons per square mile per month or per year. This terminology has led to some confusion in persuading some to believe that the measurement purports to show the deposition rate over an actual mile square area. Some have thought, too, that the selection of sampling spots should in some way be related to the square mile area unit. The fact is that the measurement indicates only the weight of dust falling out of the air at a particular location. This and other misinterpretations could be minimized if different weight-area units were employed—for example, grains per square foot. Any value expressed in tons per square mile can be converted to grains per square foot by multiplying it by 0.50.

Effects of Differing Procedures:

Among the objections to comparing dust fall in different cities are those stemming from discrepancies that arise due to varying procedures. It is a fact that there is no single widely recognized standard procedure.

In this country simple jars of varying dimensions are employed: beakers, battery jars, stainless steel or copper cylinders, wide mouth glass jars, and the like. In some cases the jars are placed

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* Presented at the Fifty-sixth Annual Meeting of the Society, June 28–July 3, 1953.

[1] Chemist and Engineering Director, respectively, Industrial Hygiene Foundation, Mellon Institute, Pittsburgh, Pa.

directly on a roof top; in others they are supported 3 or 4 ft high on a tripod or other support. In most cases liquid antifreeze is added during the winter, but in the summer the bottom of the jar is often allowed to be dry, depending in this respect only on the occurrence of rainfall.

TABLE I.—INHERENT ERROR IN A SINGLE JAR DUST FALL MEASUREMENT.

Extent to which an individual jar may deviate from the average of three identical jar exposures.

Deviation of a Single Jar Value from an Average of a 3 Jar Cluster, per cent	Number of Jars	Per cent of Total Jars
<1	30	14
1 to 5	86	39
5 to 10	58	26
10 to 15	26	12
15 to 20	17	8
20 to 25	2	1
25 to 30	2	1
>30	1	..

TABLE II.—WATER SOLUBLE SOLIDS IN DUST FALL SAMPLES FROM OAKLAND DISTRICT OF PITTSBURGH.

Water Soluble, per cent	Number of Jars	Per cent of Total Observations
>15	2	4
10 to 15	11	23
5 to 10	7	15
1 to 5	15	31
<1	13	27

Some laboratories filter the accumulated rain water from the solids and report only the insoluble solids; others evaporate and report the total solids. We have explored these variables to determine the extent to which each is responsible for variations observed in the results.

Inherent Error of a Single Jar:

To develop data indicating the reproducibility of results from a single jar, we exposed identical jars on tripods in clusters of 3, totaling 222 jars over a period of 8 months. Taking the average of the three jars as the true value, the deviation of any one jar weight could be assessed in those terms. The results are shown in Table I.

These data show clearly that inherent errors of 10 or 15 per cent are common, that errors of 15 to 20 per cent are not uncommon and that a single jar could deviate as much as 25 to 30 per cent about once in 100 cases. Those data teach us not to rely heavily on a single sample, a source of possible error that is minimized by grouping and averaging similar data, as is demonstrated later.

TABLE III.—EFFECT OF JAR ELEVATION ON DUST FALL.

Data based on 2 jars at each elevation.

Comparison of Roof Surface Dust Fall with 2-ft, 4-ft, and 7-ft Dust Fall[a]			Comparison of 2-ft, 4-ft and 7-ft Dust Falls	
$\frac{A}{B}$	$\frac{A}{C}$	$\frac{A}{D}$	$\frac{B}{C}$	$\frac{D}{C}$
1.38	1.47	...	1.07	1.06
1.25	1.47	...	1.17	1.06
1.84	1.48	...	0.80	1.10
1.39	1.28	...	0.92	...
1.83	2.08	...	1.14	...
...	1.93	1.82	...	...
...	1.69	1.53	...	...
...	1.50	1.42	...	...

[a] *A* is dust fall at roof surface, *B* is dust fall at 2-ft elevation, *C* is dust fall at 4-ft, and *D* is dust fall at 7-ft.

Water Soluble Fraction:

The magnitude of error due to loss of water-soluble materials where rain water is removed by filtration and discarded was studied for Pittsburgh dust fall. The results are presented in Table II.

These data show that the water-soluble fraction rarely exceeds 15 per cent, and demonstrates that the error resulting from the practice of discarding the filtrate is minor. In any case, a correction factor is easily estimated.

Effect of Jar Elevation:

A series of jars was positioned at different elevations above the roof top for comparison of their deposit rate with that in a jar resting directly on the flat roof surface. As shown in Table III, the jars resting on the roof receive much larger amounts of dust than those at 2 ft or greater elevation. Obviously, this is due to their sensitivity to wind-blown dust which evidently is concentrated in the air layer 10 to 12 in. above the roof surface. There appears to be no significant difference between the 2-, 4-, or 7-ft levels. We favor a level of 3 or 4 ft.

The most significant thing shown by the data is that there is no consistent ratio. The minimum ratio is about 1.3 and the maximum about 2. Therefore, dust fall data obtained from jars resting on a roof surface should be corrected by those ratios before comparison with tripod data. This is done by multiplying roof data by 0.50 and 0.75, expressing the corrected result *as a range*. For example, a roof surface value of "100 tons per sq mile per month" would be corrected for comparison with data obtained at a higher elevation, and thus described as "50–75 tons per sq mile per month."

TABLE IV.—RELATIVE[a] DUST FALL IN WET *versus* DRY BOTTOM JAR.

Range of Wet to Dry Ratios	Number of Cases	Per Cent of Total
0.85 to 1.10	32	47
1.10 to 1.30	17	25
1.30 to 1.50	13	19
1.50 to 1.70	3	4
1.70 to 2.00	3	4

[a] Clusters of three jars, one wet bottom, two dry bottom (dry except for rainy periods).

Wet Bottom versus Dry Bottom:

For studying the effect of a wet or dry bottom jar, a small amount of permanent antifreeze liquid (ethylene glycol) was placed in the bottom of one jar in a cluster of three. The other two were dry except in rainy periods. Relative values are shown in Table IV. It is apparent from these data that an appreciably greater quantity of dust may be found in a jar with a wet bottom than one with a dry bottom.

The fact that jars would normally have a wet bottom throughout the winter season due to the necessity for antifreeze indicates the logic of continuing the practice of adding a nonvolatile liquid to each jar during the summer months in order that the operations continue on the same basis the year around. If this is not done, it becomes necessary to apply a factor to the summer data to make it comparable with winter data, and these studies indicate that the factor (as in the case of jar elevation correction) would have to be dual, namely, 1 and 1.5. Thus, if the "dry" bottom data for a summer period indicated "60 tons per sq mile per month," it would be corrected to "60 to 90 tons per sq mile per month."

Effect of Jar Dimensions:

The effect of jar shape and dimensions has not been fully explored at this writing. We have a few data on the relative dust fall values obtained by exposure of the type jars indicated in the following table:

Jar Type	Diameter, cm	Ratio of Height to Diameter
1500-ml Beakers	13	1:1
Battery Jar	11	2:1
Hydrometer Cylinder	7	4:1
Gallon Preserve Jar	8	>4:1

Jars of each type were exposed in clusters of three from which an average was obtained.

The number of observations is not statistically significant and they are, therefore, not being reported at this time. It may be stated, however, that thus far we are persuaded that the dimensions of the jar openings within the range of jar sizes in common use is of little practical significance, although there is some indication of somewhat less scatter in the data obtained by the 1500-ml beakers. Differences between the various vessels in any one month are sometimes considerable, but when our 5-month data (involving 15 jars of each type) were averaged, the greatest difference between any two types did not exceed about 15 per cent.

We judge that the English deposit gage (which consists of a shallow funnel connected at the bottom to a rain water bottle) would give results differing considerably from wet bottom cylinder results, due to the permanent dry bottom of its collection surface and the relatively incomplete protection of the shallow funnel bottom against losses due to wind action.

Recommendations:

Based on the findings of these studies, it is believed that the following procedures are desirable to obtain the most consistent results at a single location. (They conform to existing practices in Chicago, Ill., New York, N. Y., Milwaukee, Wis., St. Paul, Minn., Wyandotte, Mich., New Haven, Conn.)

1. Jars should be exposed at several locations to obviate the inherent error of a single jar as well as for coverage of an area.
2. Jars should be mounted at an elevation of 2 to 4 ft above the roof top or other supporting surface.
3. The jar bottom should be permanently wet, in summer as well as winter. Permanent antifreeze liquids are most convenient. Use of water requires frequent attention.
4. If jar contents are filtered and only insoluble solids weighed, it is desirable to ascertain by special tests the characteristic percentage of water soluble matter for a particular city. In most, if not all, cases it will be found rarely to exceed 10 to 15 per cent. This correction can then be applied to all insoluble residue values thereafter.

LOCATION OF JAR STATIONS

The large particles that contribute principally to dust fall emanate from numerous sources, and this fact, combined with their falling tendencies, results in extreme variation in distribution even within small distances. Dust fall stations should be located remote from any single source of dust, and this includes not only chimneys of coal-burning furnaces on the building on which the jar may be placed and those of adjacent buildings, but also nearby horizontal roof surfaces at a greater elevation which could supply abnormal quantities of wind-swept dust to the location of the test jar.

These location specifications are often difficult, sometimes impossible to comply with. A larger number of stations tends to offset disadvantages of a particular location. In small cities attachment of the jar holder to utility poles can sometimes be arranged and, if suitably located with respect to adjacent sources, constitutes an advantageous arrangement.

Even when these specifications are met, considerable variation between two jars may be found within short distances. We have observed variations of 2 to 1 and greater between two sets of jars located within one block of each other.

TREATMENT OF DATA

The variations in dust fall in different areas of a community are usually very large and it is obviously quite meaningless to report the total dust fall or the average dust fall of a large city. For this reason, it is impossible to compare dust fall in different cities by taking averages of all the dust fall measurements in each city.

DISTRICT CLASSIFICATION

It is, however, logical and useful to classify the dust fall data in accordance with the type of neighborhood in which they were obtained. Averages of all data obtained in one kind of district can then be expected to provide a significant description. We have employed the classification below on a tentative basis:

Class A—Suburban or Agricultural

Class B—City Residential (relatively remote from industrial areas)

Class C—Residential Areas, Adjacent to Industrial Districts (including commercial districts)

Class D—Industrial District (where few people reside)

We have obtained data from a number of cities, classified in accordance with this scheme, and have grouped and averaged the data in accordance with the type of district. Comparisons on this basis provide a significant picture, with some qualifications discussed later. All figures employed, with one exception mentioned later, were obtained by methods that conform to the procedures previously outlined.

TABLE V.—WINTER AND SUMMER DUST FALL[a] CLASSIFIED BY TYPE OF NEIGHBORHOOD IN VARIOUS CITIES.

(Tons per square mile per month)

WINTER DUST FALL (JANUARY–MARCH)

	New York	Chicago	Pittsburgh	Cincinnati	Milwaukee	Wyandotte, Mich.	St. Paul	New Haven	Holyoke
Suburban	...	30	...	7 to 10	12	...	...	18	15
City residential	79[b]	84	59	11 to 17	21	20	17	18	33
Residential, near industrial or commercial	126[c] 70[d]	129	69	31 to 47	35	36	18	27	100
Industrial	222[e]	...	66	40 to 60	...	...	64	...	...

SUMMER DUST FALL (JUNE–AUGUST)

	New York	Chicago	Pittsburgh	Cincinnati	Milwaukee	Wyandotte, Mich.	St. Paul	New Haven	Holyoke
Suburban	...	23	...	14 to 21	15	...	...	7	10
City residential	33[b]	41	28	19 to 29	21	23	12	11	15
Residential, near industrial or commercial	57[c] 37[d]	65	46	31 to 47	32	37	12	13	31
Industrial	67[e]	...	51	31 to 47	...	...	50	...	...

[a] 1951 data used for N. Y., Pittsburgh, Chicago, Cincinnati, St. Paul. 1952 data for New Haven, Wyandotte, and Milwaukee. 1949 data for Holyoke.

[b] Bronx, Brooklyn, Queens.

[c] Manhattan.

[d] Brooklyn.

[e] Manhattan, Queens.

SEASONAL GROUPING

The data of a given month do not, basically, have greater significance than would those for a period of 3 weeks or $5\frac{1}{2}$ weeks, since the calendar is merely an arbitrary time subdivision. Seasons, on the other hand, are significant time periods because of the weather and fuel consumption characteristic of each. Furthermore, there is often a sharp and irregular variation in the data of consecutive months which confuses their true significance. For these reasons we recommend grouping of the data in accordance with seasons, averaging them for the total period. We have employed the following grouping:

Winter	January, February, March
Spring	April, May
Summer	June, July, August, September
Fall	October, November

Logically, winter should also include December, but in our tabulations this was not done because the custom of tabulating data by the calendar year made it inconvenient to assemble the necessary data from the different cities.

Grouping the data by season and by type of district results in a relatively large number of figures of a consistent character, and the average effectively damps out the extreme variation of an individual series.

TABLE VI.—CHARACTERISTIC DUST FALL IN AMERICAN CITIES.

(Tons per square mile per month)

Class of District	Winter	Summer
Suburban	10 to 20	5 to 15
City residential	20 to 60	10 to 30
Near industrial	20 to 125	15 to 60
Industrial	(extreme range)	

The relatively narrow range of values for comparable districts of most of the cities, especially in the suburban and city residential classes, is an indication of the value of this method of data treatment and interpretation. On the basis of these limited data, the summary in Table V has been prepared, indicating how this method facilitates city to city comparisons (see also Table VI).

The values, not surprisingly, spread over a quite wide range in the last two industrial classes.

The Cincinnati data are figures corrected by applying a factor of $\frac{1}{2}$ and $\frac{3}{4}$ to the original data reported, since their measurements are made by jars resting directly on roof surfaces (see previous discussion on Effect of Jar Elevation).

CLASSIFICATION SYSTEM

The district classes employed herein have one advantage: the titles appear to define them clearly, since in our correspondence with cooperating city officials no questions were raised concerning their meaning. However, we are by no means satisfied that there was in fact a common understanding of them and that all stations in the different cities are grouped by the same standard. Basic differences in the characteristics of cities make for some difficulties, unless the definitions are made completely explicit.

Some cities by their nature will have no suburban areas. Some have industrial districts with practically no dwelling houses; others with light industry may have a considerable population within the industrial district.

The grouping of commercial areas with dwelling areas adjacent to industrial districts, mainly to keep the system simple, is open to question.

The principle of classification is the thing of importance. Each city should feel free to make such modifications in the classification system as may be dictated by its particular characteristics to result in a more realistic presentation.

If a station or stations within a group show figures consistently much higher or lower than the average, that location should be re-examined from these viewpoints: (*a*) Is it unduly affected by a known nearby source, and therefore is it advisable to move or eliminate it? (*b*) Can justification be made, by reference to fundamental definitions, for changing its classification for averaging purposes?

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON PORCELAIN ENAMELS AND CERAMIC COATINGS AS ENGINEERING MATERIALS

The Symposium on Porcelain Enamels and Ceramic Coatings as Engineering Materials, sponsored by Committee C-22 on Porcelain Enamel, was held at the Twenty-sixth and Twenty-eighth Sessions of the Fifty-sixth Annual Meeting of the Society held in Atlantic City, N. J., on July 2, 1953.

Because of recent developments in the porcelain enamel field, Committee C-22 felt that a symposium would be of interest to many engineers now interested with the work but not associated with the porcelain enamel or ceramic coating fields. The symposium presented data on the mechanical properties of these materials and indicated the trend of future developments. It included the following papers:

Introduction—Dwight G. Bennett
Some Examples of the Functional Use of Porcelain Enamel and Ceramic Coatings for Steel—G. H. Spencer-Strong
Resistance of Porcelain Enamels to Weathering—Dwight G. Moore
The Chemical Resistance of Glass Fused to Steel—E. A. Sanford and O. J. Britton
Acid Resisting Properties of Porcelain Enamels—Harold C. Wilson
Requirements for and Expected Benefits from the Application of Coatings to High-Temperature Components of a Jet Engine—A. C. Francisco and G. M. Ault
High-Temperature Ceramic Coatings as Applied to Aircraft Power Plants—B. L. Paris
The Industrial Processing of High-Temperature Ceramic Coatings—J. H. Terry
The Abrasion Resistance of Various Types of Porcelain Enamel—Arthur V. Sharon
The Resistance of Porcelain Enamels to Surface Abrasion as Determined by the PEI Test—John T. Roberts
Torsion Testing as an Aid to the Porcelain Enamel Industry—E. L. Hoover
The Strengthening Effect of Porcelain Enamel on Sheet Iron as Indicated by Bending Tests—E. E. Bryant
Tension Tests of Porcelain Enameled Steel—W. A. Deringer
Effect of Temperature on the Electrical Resistivity of Several Ceramic and Silicone-Type Coatings—Simon W. Strauss, Lloyd E. Richards, and Dwight G. Moore
A Laboratory Evaluation of Ceramic Coatings for High-Temperature Applications—Sara J. Ketcham
Guideposts in Selecting Porcelain Enamels and Ceramic Coatings—A Summary—W. N. Harrison

These papers, with discussions, are issued as *ASTM Special Technical Publication No. 153* entitled "Symposium on Porcelain Enamels and Ceramic Coatings as Engineering Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON LATERAL LOAD TESTS ON PILES

The Symposium on Lateral Load Tests on Piles, arranged by Committee D-18 on Soils for Engineering Purposes, was held at the Twentieth and Twenty-third Sessions of the Fifty-sixth Annual Meeting of the Society in Atlantic City, N. J., July 1, 1953. Committee D-18 hopes that the material presented at this symposium will lead to the development of standard procedures for making lateral load tests on piles.

The following papers were presented:

Introduction—A. E. Cummings
Resistance of Long Hollow Piles to Applied Lateral Loads—G. A. McCammon and J. C. Ascherman
Lateral Load Tests on Groups of Battered and Vertical Piles—Lawrence B. Feagin
Bearing Piles Subjected to Horizontal Loads—L. T. Evans
The Resistance to Lateral Loading of Single Piles and of Pile Groups—Gregory P. Tschebotarioff
The Lateral Load Capacity of Timber Pile Groups—J. O'Halloran
Lateral Load Tests on Vertical Fixed-Head and Free-Head Piles—Sol M. Gleser
Lateral Load Tests on Piles for Design Information—A. A. Wagner

The papers and discussions are issued as *ASTM Special Technical Publication No. 154* entitled "Symposium on Lateral Load Tests on Piles."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON DYNAMIC TESTING OF SOILS

The papers and discussions in the Symposium on Dynamic Testing of Soils were presented at the Twenty-ninth and Thirtieth Sessions of the Fifty-sixth Annual Meeting of the Society held on July 2, 1953, in Atlantic City, N. J. Committee D-18 on Soils for Engineering Purposes sponsored this symposium with two main purposes in mind, namely, to assemble, for publication, all available information and to discuss as yet unsolved problems in the field of soil dynamics.

The following papers were presented:

Introduction—R. K. Bernhard
Elasticity and Damping of Oscillating Bodies on the Soil—H. Lorenz
The Pressures Generated in Soil by Compacting Equipment—A. C. Whiffin
Loose Sands—Their Compaction by Vibroflotation—Elio D'Appolonia
Performance Records of Engine Foundations—Gregory P. Tschebotarioff
Macromeritic Liquids—Hans F. Winterkorn
Compaction of Sand at Resonant Frequency—Frederick J. Converse
The Elastic Theory of Soil Dynamics—Patrick M. Quinlan
Pilot Studies on Soil Dynamics—R. K. Bernhard and J. Finelli
A Dynamic Analogy for Foundation-Soil Systems—Adrian Pauw
A Discontinuous Model for the Problems of Soil Dynamics—J. J. Slade, Jr.
Vibration Research on Road Constructions—C. van der Poel
Vibrations in Semi-Infinite Solids Due to Periodic Surface Loading—Tse Yung Sung
Soil Dynamics Bibliography—R. K. Bernhard

This Symposium is issued as *ASTM Special Technical Publication No. 156* entitled "Symposium on Dynamic Testing of Soils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON FLUORESCENT X-RAY SPECTROGRAPHIC ANALYSIS

In recent years, improvements in the generation of high-intensity X-ray beams and in the means of detecting and measuring X-rays have made fluorescent X-ray spectrographic analysis feasible. Its practical value has been attested to by the numerous papers published in the past five years.

The papers in this Symposium on Fluorescent X-ray Spectrographic Analysis were presented at the Fourth Session of the Fifty-sixth Annual Meeting of the Society held in Atlantic City, N. J., June 29, 1953.

This symposium, which was sponsored by Committee E-2 on Emission Spectroscopy, included the following papers:

Introduction—E. B. Ashcraft
Basic Theory and Fundamentals of Fluorescent X-ray Spectrographic Analysis—H. Friedman, L. S. Birks, and E. J. Brooks
The Correlation Between Fluorescent X-ray Intensity and Chemical Composition—J. Sherman
Use of Multichannel Recording in X-ray Fluorescent Analysis—M. F. Hasler and J. W. Kemp
Examination of Metallic Materials by X-ray Emission Spectrography—R. M. Brissey, H. A. Liebhafsky, and H. G. Pfeiffer
An Absolute Method of X-ray Fluorescence Analysis Applied to Stainless Steels—Gordon E. Noakes
The Fluorescent X-ray Spectrographic Analysis of Minerals—Howard F. Carl and William J. Campbell

The papers are issued as *ASTM Special Technical Publication No. 157* entitled "Symposium on Fluorescent X-ray Spectrographic Analysis."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIOACTIVITY—ISOTOPES AND TRACER TECHNIQUES

The papers in this symposium were presented at the Seventh Session of the Fifty-sixth Annual Meeting of the Society held on June 30, 1953, in Atlantic City, N. J.

The following papers are published as *ASTM Special Technical Publication No. 159* entitled "Symposium on Radioactivity—Isotopes and Tracer Techniques."

Introduction—Don M. McCutcheon
Properties and Uses of Radioisotopes—G. D. Calkins
Applications of Radioactive Measurements to ASTM Work—O. M. Elliott and J. W. Kuranz
Design of Radioisotope Laboratories—G. G. Manov and O. M. Bizzell
Training Personnel in Radioisotope Techniques—Donald R. Smith
Instrumentation—E. H. Wakefield
Management Problems Resulting from Radioisotope Utilization by Industry—W. L. Davidson

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LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been printed during the year 1953 and issued as separate publications not included in either Proceedings or ASTM BULLETIN. For a list of earlier Special Technical Publications, see the original listings in Proceedings, Volume 48, page 1352, and the supplementary listings in Volume 49, page 1240, Volume 50, page 1490, Volume 51, page 1322, and Volume 52, page 1309. A complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50 Year Index to ASTM Publications.

Special Technical Publication Number	Title
41-C	Index to the Literature on Spectrochemical Analysis, Part III, 1946–1950 (1953)
150	Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (1953)
151	Report on the Elevated-Temperature Properties of Chromium-Molybdenum Steels (1953)
152	Symposium on Insulating Oils, Fifth Series (1953)
153	Symposium on Porcelain Enamels and Ceramic Coatings as Engineering Materials (1953)
154	Symposium on Lateral Pile Load Tests (1953)
155	Symposium on Techniques for Electron Metallography (1953)
156	Symposium on Dynamic Testing of Soils (1953)
157	Symposium on Fluorescent X-Ray Spectrographic Analysis (1953)
158	Symposium on Effect of Temperature on the Brittle Behavior of Metals with Particular Reference to Low Temperatures (1953)
159	Symposium on Radioactivity—Isotopes and Tracer Techniques (1953)